

ORDOVICIAN BENTONITES FROM ONTARIO

BY

JOHN BARRIE BAIN ORR B.Sc.

MAY, 1959.

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THE UNIVERSITY OF ALBERTA

"ORDOVICIAN BENTONITES FROM ONTARIO"

A DISSERTATION

SUBMITTED TO THE FACULTY OF GRADUATE STUDIES

IN PARTIAL FULFILMENT OF THE REQUIREMENTS FOR THE DEGREE

OF MASTER OF SCIENCE

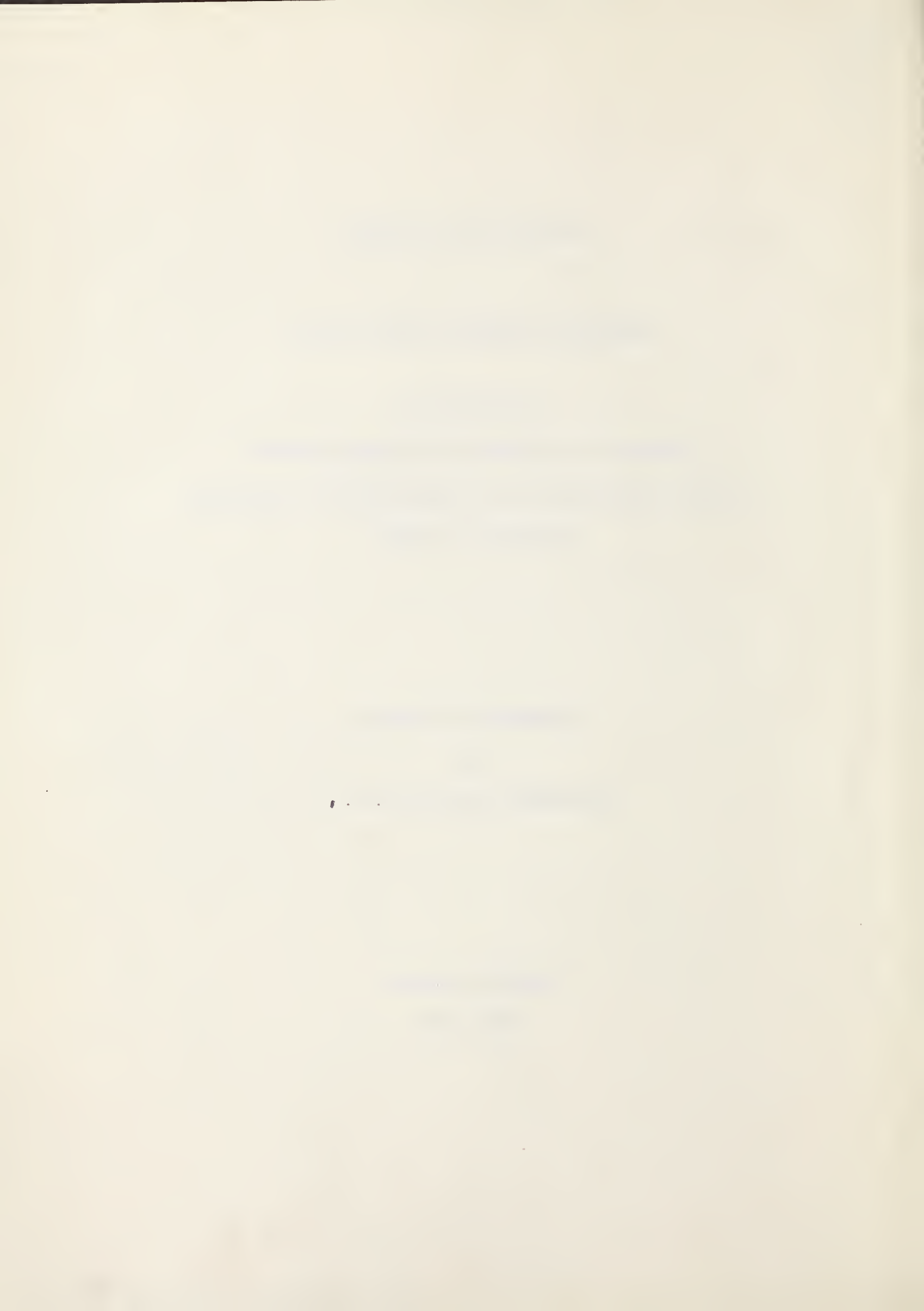
DEPARTMENT OF GEOLOGY

by

JOHN BARRIE BAIN ORR B.Sc.

EDMONTON, ALBERTA

MAY, 1959



ABSTRACT

Five samples of Ontario Ordovician bentonite were examined by using the techniques of heavy and light mineral separation, x-ray diffraction patterns, zircon studies, and base exchange analysis. The clay fraction of the bentonite consists of a mixed-layer aggregate dominated by illite but with some montmorillonite present. In addition, small amounts of chlorite were noted.

The mineral assemblage indicates that the clay altered from a volcanic glass with the composition of a latite. Similar bentonites in Ordovician strata throughout the eastern United States can be traced along with the Ontario horizons to a volcanic vent in the crystalline region of North Carolina.

Potassium-argon age dating yielded tentative ages of 656 and 654 m.y. on feldspars, and 260 m.y. on volcanic glass. This suggests contamination of feldspar and leakage of argon from the volcanic glass. It is felt that future runs on the remaining extracted argon samples may produce more satisfactory results.



ACKNOWLEDGEMENTS

The writer wishes to express his sincere appreciation to all members of the Department of Geology, University of Alberta, for their helpful suggestions and encouragement. Thanks are especially due to Dr. R. E. Folinsbee, under whose direction and guidance this thesis was written. Dr. H. Baadsgaard provided competent assistance in the base exchange analysis and potassium-argon age determinations, as did Dr. J. F. Lerbelmo in the mineralogical studies. Guidance was obtained from Dr. F. A. Campbell in the x-ray analysis.

Technical assistance was rendered by Dr. L. Halferdahl of the Research Council of Alberta, and F. Dimitrov of the Department of Geology. Miss S. Baker typed the manuscript.



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CHAPTER ONE

INTRODUCTORY

Introduction

Ordovician sedimentary strata of Central Ontario contain a number of altered volcanic ash beds. Good exposures of these horizons may be found in the numerous abandoned limestone quarries north of Lake Simcoe. Samples were collected from three of the bentonite beds for the primary purpose of determining their absolute age.

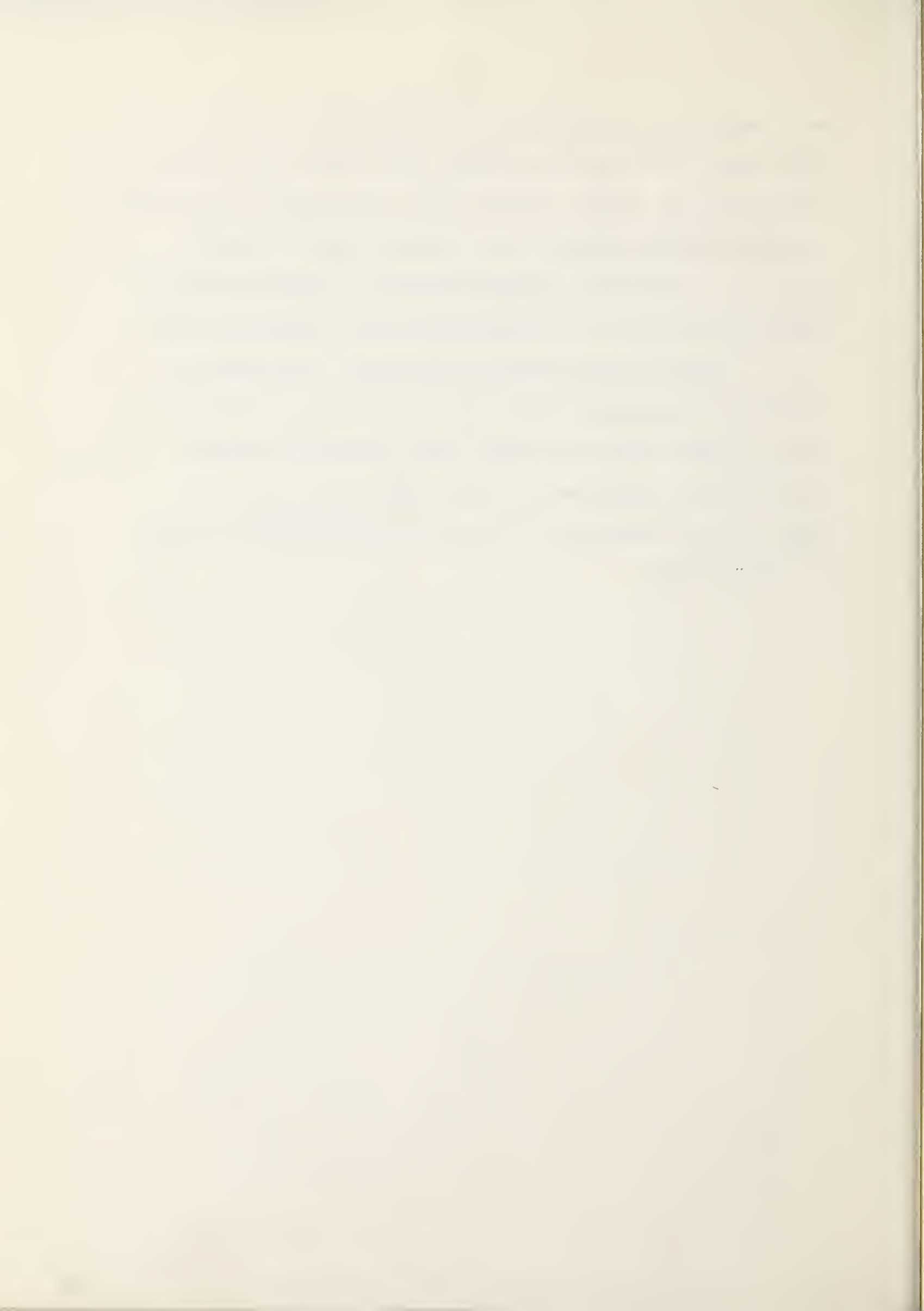
Bentonites with similar physical characteristics and stratigraphic position have been noted and studied throughout the eastern United States and Sweden. Consequently, heavy mineral studies, with emphasis upon the zircon content, light essential mineral analysis, base exchange and x-ray studies were undertaken on the Ontario bentonite with the aim of comparing and contrasting the properties of the bentonites in different regions. A direct correlation was found between the Ontario and United States volcanic material. Some differences were noted in comparisons of the Ordovician bentonites from Sweden, with those from the thesis area.

Previous Work

Very little information concerning the bentonite horizons in Ontario could be found in the literature. Maddox (1930) noted volcanic material in well cuttings near Collingwood, Ontario. C. S. Ross of the United States Geological Survey analysed the sample and found it similar to Ordovician bentonites in Virginia. More recently, Liberty and Caley (1950, 1952) and Liberty (1953)

noted bentonite partings in limestone of Black River age in the thesis area. It is from these horizons that samples were taken for the thesis study. Forman and Lake (1954) analysed the clay constituents of these bentonites and came to the conclusion that the clay was composed of a mixed-layer aggregate dominated by illite but with some montmorillonite present. The writer has formed a similar opinion.

Numerous studies have been undertaken on the Ordovician bentonites of the eastern United States. The reader is referred to papers by Nelson (1922), Ross (1928, 1955), Allen (1929, 1932), Kay (1931, 1935, 1944), Fox and Grant (1944), Weiss (1954) and Weaver (1953). Bentonites in Sweden have been described in detail by Byström (1955).



CHAPTER TWO

STRATIGRAPHY

The Ordovician strata of the thesis area, that is the region north of Lake Simcoe, Ontario, lie unconformably on Precambrian granite and granite-gneiss. They are divided into eight lithologic units, the upper two being absent in some areas, (Table I).

BLACK RIVER

The lowermost "Basal" beds consist of green shale and red, sandy shales. They are not present everywhere due to the uneven nature of the Precambrian topography. The "Basal" beds are unfossiliferous and their maximum thickness attains 40 feet.

Resting on the "Basal" beds are 30-40 feet of limestone comprising the Pamela beds. The limestone varies from brown to greenish grey and may be divided into three lithologic units on the basis of color and grain size. Chert nodules and glauconite coatings on fossils and enclosed pebbles are present. The upper few feet changes laterally eastward from a fine-grained to lithographic, brown limestone into a pale green and greenish grey, fine-grained dolomitic limestone.

The succeeding Lowville beds consist of some 35 feet of grey to brown and cream lithographic limestone. The lower unit is comprised of thinly laminated, grey, lithographic limestone of varying thickness. Green argillaceous partings are common and near the base is the Mx bentonite horizon (Liberty, 1958). The uppermost 10-12 feet appear more massive and contain the coral species Tetradium cellulorum and Tetradium fibratum. The base of this unit

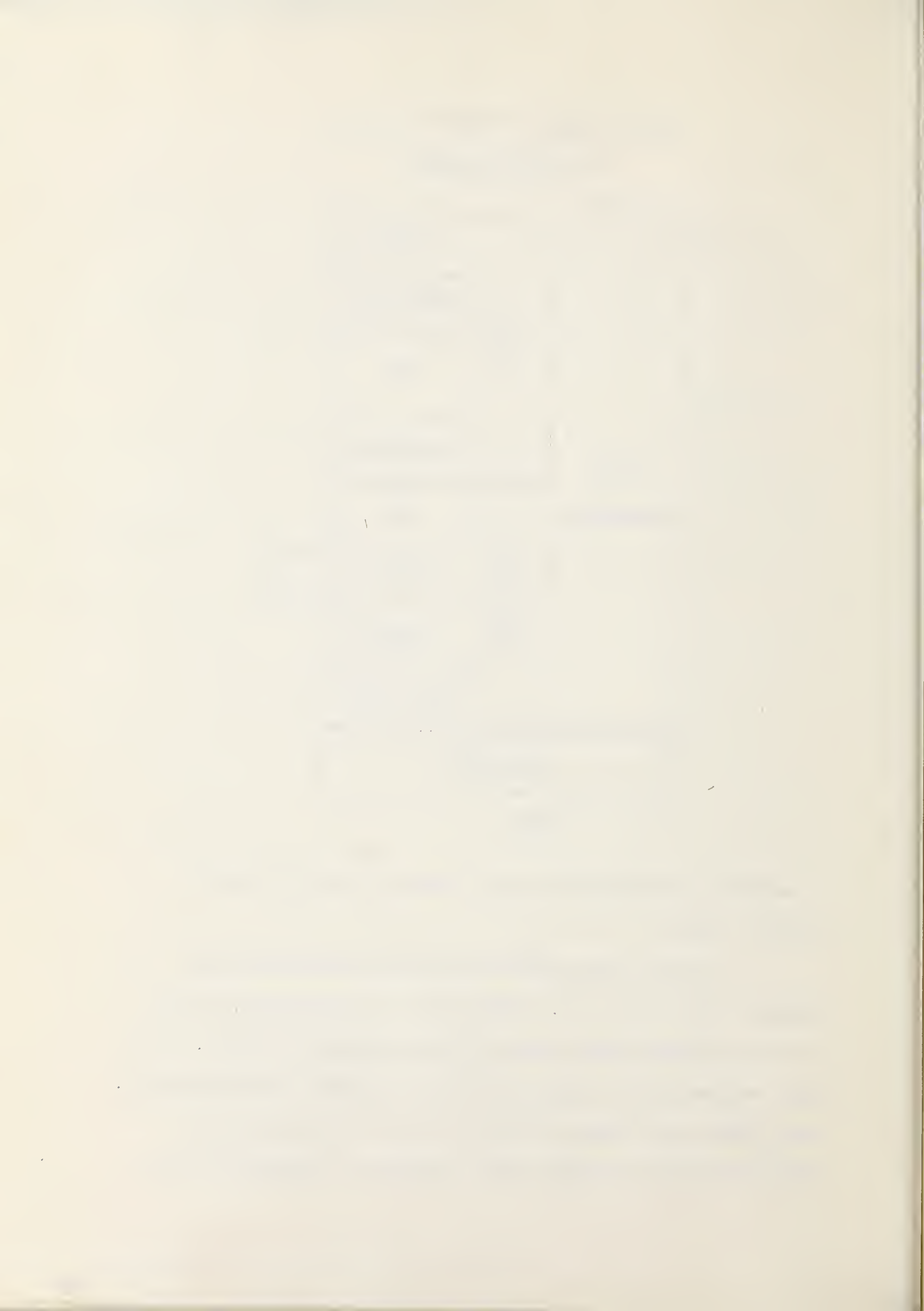
COLUMNAR TABLE OF ORDOVICIAN STRATA
IN SOUTHERN ONTARIO

MIDDLE ORDOVICIAN	TRENTON	COBCURG	
		SHERMAN FALL	
		HULL	
		ROCKLAND	
	BLACK RIVER	LERAY	
		LOWVILLE	-----M _H
		PAMELIA	-----M _x
		BASAL BEDS	
		PRECAMBRIAN	

TABLE I

is marked by a one-half inch seam of bentonite; the M_H horizon (Liberty, 1958).

The Leray beds overlie the Lowville and constitute the uppermost Black River unit. They consist of approximately 17 feet of grey and brownish grey, fine and medium-grained limestones. Some grey, medium-grained, fragmental (clastic) limestone beds are present. Chert nodules are common near the top of this unit which is also characterised by an abundant faunal assemblage including the corals



Lyopora (= Columnaria), Streptelasma, and Calapoecia; the brachiopod Rafinesquina; the stromatoporoid Stromatoceria; and several genera of nautiloids.

TRENTON

Strata of Trenton age overlie the Black River units with apparent conformity; but a definite physical change occurs at the top of the Leray beds, the succeeding strata being in general coarser textured and denser. The change seems to coincide with a decrease in fossil content. The Trenton beds are divisible into four lithological units which are in ascending order; the Rockland, Hull, Sherman Fall, and Cobourg beds.

The Rockland-Hull beds, which have a normal thickness of about 55 feet are composed of grey and brownish grey, fine-and medium-grained limestone and crystalline limestone. The brachiopod genus Dalmanella is common.

Sherman Fall and Cobourg beds are not present everywhere in the area. Where exposed, the Sherman Fall strata consist of some 170 feet of light to dark grey, fine-grained, argillaceous limestone, with interbeds of crystalline limestone.

Cobourg beds succeed the Sherman Fall, and constitute the youngest strata in the area. They comprise approximately 185 feet of grey to brownish, fine-grained and dense limestone and argillaceous limestone. Fossils are common and are represented by the brachiopods Cyclospira bisulcata and Rafinesquina deltoidea, and the gastropods Hormotoma trentonensis and Trochonema umbilicatum.

A mantle of glacial drift covers the area and largely

conceals the bedrock so that the best exposures of Ordovician strata are found in the numerous quarries. Samples of bentonite were obtained from four of these quarries and represent three different ash falls.

The strata are generally flat lying although dips up to 22 degrees have been noted near the border of the Canadian Shield.

The foregoing discussion on the stratigraphy of the thesis area is by no means all inclusive. It is merely intended to give the reader a general idea of the geologic setting associated with the bentonite horizons studied. For a more detailed description of the Ordovician strata of the area, the reader is referred to the various reports by Caley and Liberty (1950, 1952) and Liberty (1953) of the Geological Survey of Canada.

Occurrence of Ordovician Bentonites in North America

Bentonites of Ordovician age, commonly termed metabentonite or K-bentonite, are to be found over a widespread area in eastern North America (Figure 1). They occur in the Lowville and Chaumont formations of the Black River group, and in the Rockland, Hull, and Sherman Fall Formations of the Trenton group (Kay, 1935). Metabentonite has been recognized in Kentucky, Tennessee, Alabama and Virginia by Nelson (1922); in Pennsylvania by Bonine and Holmes (1929), and by Weaver (1953); near St. Louis, Missouri by Allen (1929); in northeastern Iowa and near-by parts of Wisconsin and Minnesota by Allen (1929, 1932); and in New York by Kay (1931). These ash materials consistently become coarser to the southwest (Ross, 1953). A five foot bed of bentonite reported in beds of Lowville age in Kentucky gives good evidence of the large volume of pyroclastics ejected during this time.

LOCATION
OF
ORDOVICIAN BENTONITES

(AFTER ROSS, 1955)



FIGURE 1

Definition of Bentonite

The following definition of bentonite proposed by Ross and Shannon (1926) is generally accepted. "Bentonite is a rock composed essentially of a clay-like mineral formed by the devitrification and the accompanying chemical alteration of a glassy igneous material, usually a tuff or volcanic ash". The Ordovician bentonites studied in this analysis essentially conform to this definition. However, Ross and Shannon go on to describe the bentonite further and state that, "the characteristic clay mineral is montmorillonite". This was definitely found to be untrue in the Ontario bentonites and thus, although the term bentonite will still be applied to these rocks, it is with the understanding that the samples described do not strictly conform to the original definition.

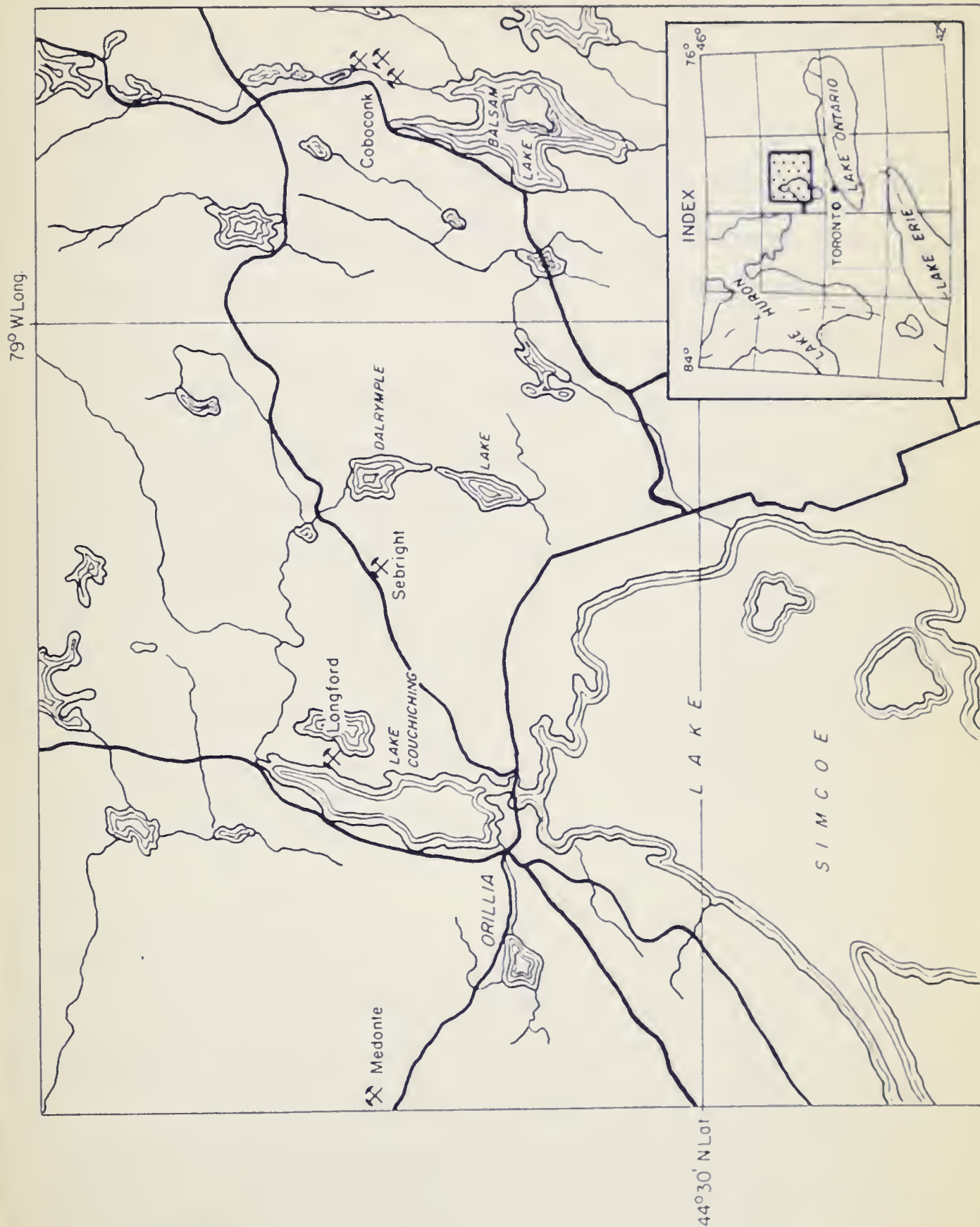
Location and Description of Samples

The samples used in this study were collected by Dr. R.E. Folinsbee during the summer of 1958. They were taken from four separate quarries located in an east-west line north of Lake Simcoe, Ontario.

Sample locations are indicated by quarry name on the index map (Figure 2), while stratigraphic position and correlations are shown on the correlation chart (Figure 3).

Petrographic descriptions were not undertaken since the bentonite was generally unconsolidated.

The color of each sample was checked against the Rock Color Chart distributed by the National Research Council, Washington, D.C. (1948).



LOCATION OF SAMPLES, LAKE SIMCOE AREA, ONTARIO

FIGURE 2



Sample #1 Medonte Lower M_H (3370)

Location: Sample taken from a quarry near the town of Medonte, Ontario.

Description: The bentonite occurs in two 1" seams which correlate stratigraphically with the M_H seam of Liberty. The two seams are separated by 3" of birdseye limestone and are overlain and underlain by limestone. A sample from the lower seam was used since the upper appeared contaminated by glacial sand. In color, the bentonite is a light olive grey (5 y 5/2). It is composed mainly of a mixed-layer aggregate clay mineral, believed to have formed from the devitrification of volcanic glass. The grit fraction contains a combination of carbonate rock fragments, (probably fallen down from the limestone above), devitrified volcanic glass, quartz, feldspars and a heavy mineral assemblage characteristic of volcanic rocks. The grit fraction is estimated at approximately 5 per cent of the sample. This sample appeared to have the largest swelling power of the five; although no quantitative determinations were made on this property.

The above general description of the composition of the bentonites holds for the other samples also, except where otherwise noted.

Sample #2 Medonte Mx

Location: Sample taken from near an old crusher on floor of the Medonte quarry.

Description: The bentonite layer is 1" thick and is overlain by limestone and underlain by crystalline dolomite. In color, the sample is pale olive (10 y 6/2). When dampened with water



it is quite sticky and plastic, but has an intermediate swelling power compared with the other samples. There is some contamination of the sample by glacial sand and gravel as evidenced by field observations and heavy mineral studies. This bentonite bed correlates with the Mx horizon of Liberty.

Sample #3 Coboconk

Location: Sample taken from the west quarry at the town of Coboconk, Ontario.

Description: This bentonite occurs over a 6" zone interbedded with limestone. An actual bentonite rich layer from which the sample was taken is only 0.5" thick although bentonite probably occurs throughout the whole zone. This sample does not correlate with either the M_H or Mx horizons of Liberty but is located 8½ feet above the M_H seam. The actual M_H seam was not well enough exposed to provide a certain sample. The sample is pale olive (10 y 6/2) in color and is slightly more consolidated than either of the Medonte bentonites. It appears to have an intermediate swelling power.

Sample #4 Sebright M_H

Location: Sample taken from an abandoned quarry approximately 25 miles southwest of the town of Sebright on the road between Sebright and Rathburn.

Description: The bentonite seam is 1" thick and occurs 2½ feet above a green shale parting. Limestone lies above and below the seam which correlates with the M_H horizon of Liberty. The sample is yellowish grey (5 y 8/1) in color and slightly consolidated. The swelling power is intermediate.



Sample #5 Longford M_H

Location: Sample taken from a quarry north of the Longford Mills.

Description: This sample appears to be more of a bentonitic shale than a pure bentonite. It is more consolidated than any of the others and has a slightly fissile habit. In color, it is greenish grey (5 GY 6/1). The sample was taken from the upper of two 1" bentonitic shale partings which appear to correlate approximately with Liberty's M_H horizon. This bentonitic shale has the lowest swelling power of the five samples analysed.

CORRELATION OF "BENTONITE"

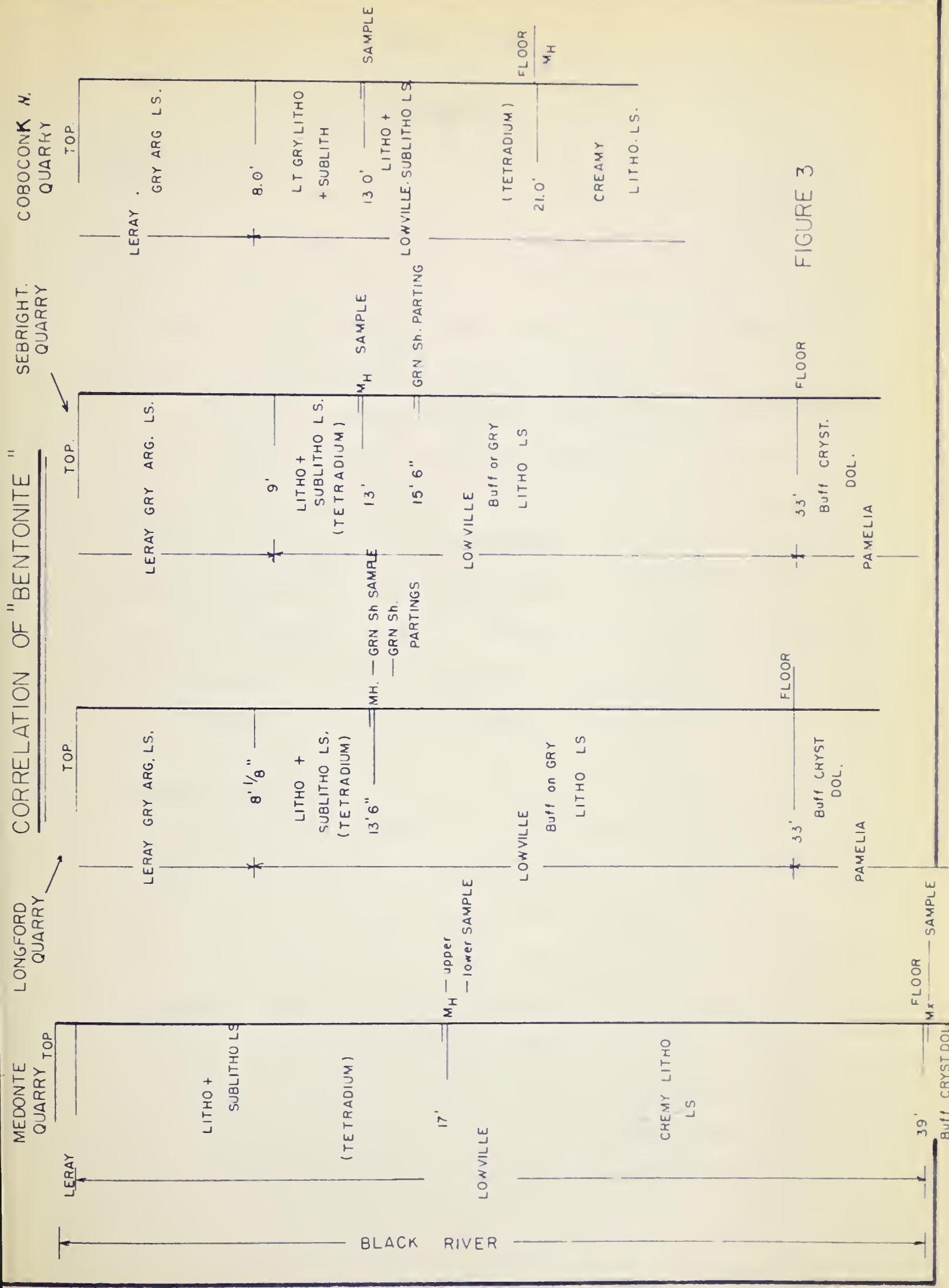


FIGURE 3



CHAPTER THREE

MINERALOGY

General Statement

Light mineral separations were carried out primarily for the isolation of feldspars which were subsequently used for K/A age dating. Optical properties of the feldspars were analysed in an attempt to identify the varieties present. A cursory examination of the other light minerals was also undertaken.

The zircon concentrate was of primary interest in the heavy mineral assemblage. A more detailed description and a statistical analysis were carried out on this mineral. A study of the remaining "heavies" was undertaken to provide another correlative tool for comparing the Ontario bentonites with those of the Eastern United States and of Kinnekulle, Sweden. The variety and types of heavy minerals present also gave an indication of the amount of contamination of the sample by detrital material.

Preparation of Material

The bentonite samples were unconsolidated and required no mechanical crushing. The samples were soaked in water for several hours to cause further disaggregation, and then mixed in a Waring Blendor, small quantities at a time. The blendor completely dispersed the bentonite giving a thick clay suspension. This suspension was subjected to a series of decantations with water; the clay material being decanted off while the heavier "grit fraction" settled to the bottom. The clay fraction was collected in one large vessel and the clean "grit" was concentrated in a beaker. No dispersing agent was

added in order not to expose the clay fraction to any chemical agent which could cause ion exchange.

The "grit fraction" was then wet sieved through U. S. standard meshes, 35, 60, 80, 100, 170, 270, 325. Permanent mounts were made of the finer grain sizes to determine which grades had the largest concentrations of heavy and light minerals. The main mineral concentration was found in the -170 +270 mesh fraction, so this size grade was used for the separation of both heavies and lights. A centrifuge and tetrabromethane (S.G. 2.96 at 20° C.) were employed to separate the lights and heavies. Centrifuging at top speed for a few minutes was sufficient to separate the two fractions, which were subsequently washed several times with acetone to remove any tetrabromethane adhering to the particles.

Isolation of Feldspars

A Frantz Isodynamic separator and heavy liquids were employed to separate the feldspars from quartz, carbonate rock fragments, clay fragments and devitrified volcanic glass. A liquid with specific gravity of approximately 2.54 was made by mixing acetone and tetrabromethane. The specific gravity was checked with a pure microcline crystal. Centrifuging in this liquid resulted in separation of the quartz and carbonate fragments from the clay mineral aggregates, feldspars and devitrified volcanic glass. A magnetic separation at 1.6 amps was then employed to remove the feldspars from the volcanic glass and clay particles. The isodynamic separator was adjusted to a slope of 15 degrees and tilt of 10 degrees for all the magnetic separations. A reasonably pure feldspar sample was finally obtained.

Isolation of Zircons

The heavy mineral assemblage contained a very high percentage of pyrite. To remove this, the sample was heated at 500° C. for 5 minutes to oxidize the iron sulfide to a magnetic form. A hand magnet was employed to extract the highly magnetic material. The remaining fraction was run through the magnetic separator at 1.6 amps removing most of the other heavy minerals as well as the remaining pyrite. Further separation of the non-magnetic fraction was accomplished with methylene iodide ($C_2H_2I_2$; S.G. 3.3) in a standard two funnel separation apparatus. The zircons (S.G. 4.6-4.7) sink in methylene iodide while most other minerals float. All fractions were washed with acetone and a permanent arochlor (Index 1.66) mount prepared for each. The fraction, S.G. > 3.3, contained the main zircon concentration while apatite was prominent in the fraction lighter than S.G. 3.3.

Description of Heavy Minerals

The preponderance of opaque heavy minerals, in some cases up to 99 per cent of the total heavy assemblage, made statistical counts of the whole heavy mineral suites impractical for all samples except the Medonte M_H which contained only 80 per cent opaques. Two hundred non-opaques were counted from the original separation in this case. For the remaining samples it was necessary to remove the main concentration of opaque minerals in order to isolate a sufficiently large fraction of non-opaques. Thus, in the abundance table the original percentages of total opaques and total non-opaques are given, and then the relative abundances of the various non-opaques is presented. However, most of the zircon concentrate had been removed to facilitate the zircon

studies before the heavy mineral analysis was undertaken. Therefore, the zircon abundance had to be estimated. Much of the apatite also separated out with the zircon (non-magnetic at 1.6 amperes) and its abundance was also estimated. It was felt that these two minerals would each account for at least 10 per cent of the non-opaque heavies as is shown in the Table of Abundances (Table 2) since they are both common minerals in acid volcanic rocks.

Opagues:

Pyrite - Occurs most commonly as irregularly shaped clusters of cubic, dodecahedral or octahedral crystals and in some cases as perfectly formed individual cubic crystals. Red ferruginous staining was common on the pyrite; and in the Coboconk sample, covers practically all the grains, (some rounded grains were noted in the Medonte M_H sample). The pyrite is believed to be authigenic with the exception of the rounded grains.

Magnetite - Present in the Medonte M_H sample only, as irregularly shaped grains.

Ilmenite - Present in the Medonte M_H sample only, as irregularly shaped grains similar to the magnetite but identified by the presence of leucoxene alteration products.

Non-Opagues:

Apatite - Occurs as euhedral crystals, often elongated, prismatic, terminated by a pinacoid (001) or pyramid. Inclusions, varying in size and shape, but parallel to the c -axis are common. Some grains are slightly rounded and etched, while others appear to be broken fragments of euhedral crystals. Photomicrographs of various apatite crystals are shown in Plate 5.

Biotite - Present as pale rusty yellow to rusty brown, angular to subangular flakes. Abundant dark inclusions (carbonaceous?) were noticed in most grains.

Epidote - Occurs in the Coboconk sample only, as a single yellowish-green rounded grain. This mineral probably represents contamination.

Garnet - Occurs as very pale pink to colorless with a pale blue tinge, sharply angular grains. Conchoidal fracture is common and some fragments exhibit dark inclusions or a pitted surface.

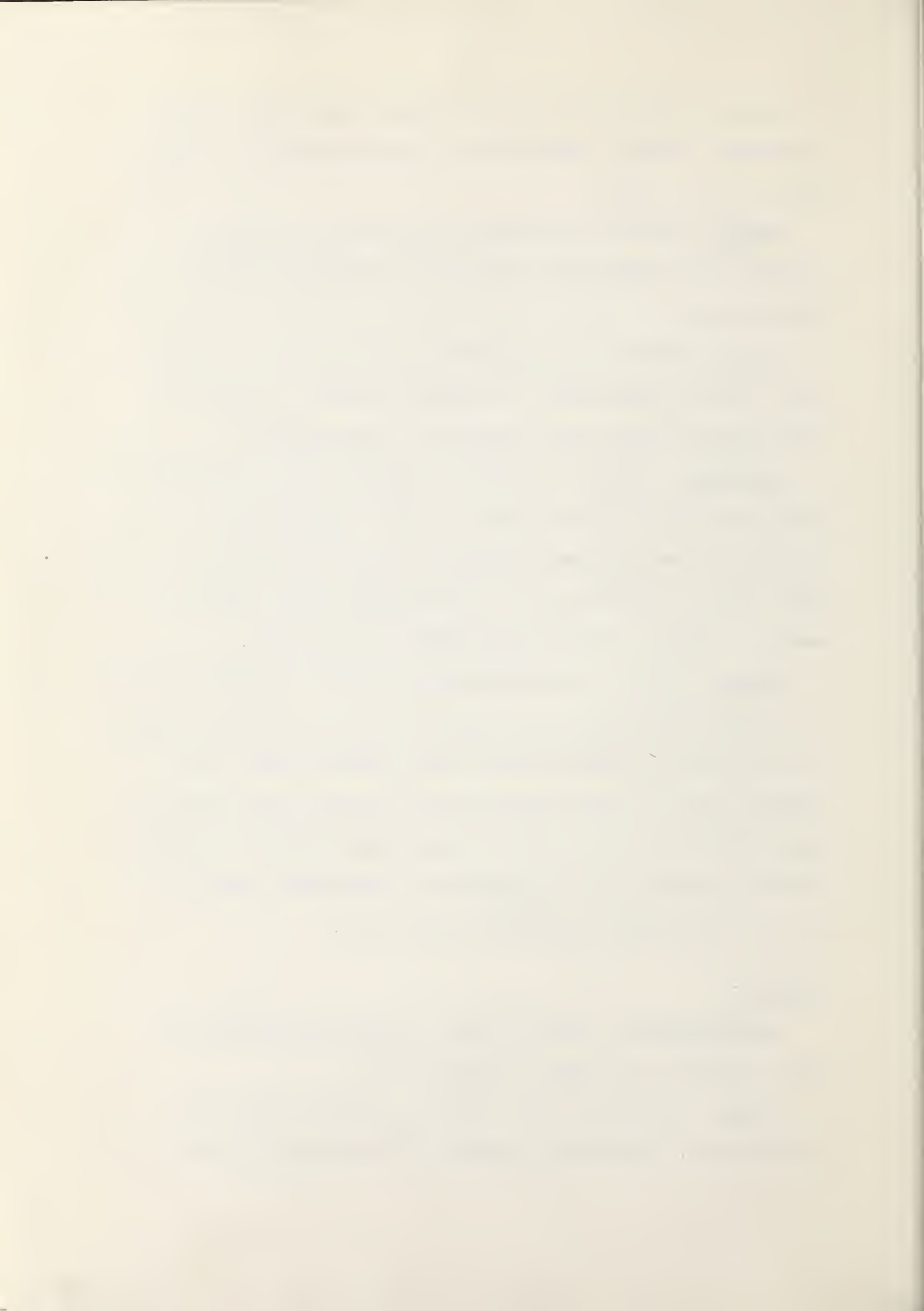
Hornblende - Present in a number of shades of green and brown. Pleochroism varies from very strong to weak. Some dark green varieties are nearly opaque, transmitting light only through the thinner edges of the grain. All the hornblende appears fresh and angular. Prismatic cleavage is noticeable in some grains.

Kyanite - Occurs as colorless elongate grains of marked rectangular outline with conspicuous cross cleavage. Only two grains were noted, one in the Medonte M_H and the other in the Coboconk sample. The Coboconk grain was larger than the other associated heavies and it is thought that its presence is due to contamination incurred during the mechanical analysis. The incongruence of a metamorphic mineral in a volcanic suite further substantiates this belief.

Pyroxenes:

Aegerine-Augite - Occurs as angular fragments generally light green pleochroic to a lighter yellowish-green.

Augite - Occurs as angular fragments of a medium green color, non-pleochroic. Dark-brown inclusions or alterations are common and



some etching of the grains was observed.

Diopside - Occurs as very pale green to colorless angular fragments. Some grains are slightly etched.

Hyperthene - Occurs as medium-to light-green angular fragments, pleochroic to pinkish-brown or pale yellow.

Rutile - Present as rusty-red to rusty-yellow angular fragments or euhedral crystals.

Sphene - Occurs as a clear, irregular, angular fragment exhibiting conchoidal fracture. One grain only was noted, in the Coboconk sample.

Tourmaline - Occurs as angular fragments for the most part, but the Medonte Mx sample has two rounded bluish-green grains. The color generally varies from medium blue to medium brown. Small dark inclusions may or may not be present.

Zircon - Zircon is described in detail under "Zircon Studies".

Zircon Studies

A study of the zircon concentrates of the five samples analysed shows a close similarity in varieties present. These may be broken into four general groups:

1. euhedral crystals with sharp prismatic terminations
2. euhedral crystals with slightly rounded prismatic terminations
3. broken fragments of crystals, generally angular
4. distinctly rounded grains.

The characteristics of the euhedral crystals are similar for both the sharply angular and slightly rounded varieties. This rounding could be largely due to magmatic corrosion as postulated by

TABLE 2. RELATIVE ABUNDANCE OF HEAVY ACCESSORY MINERALS

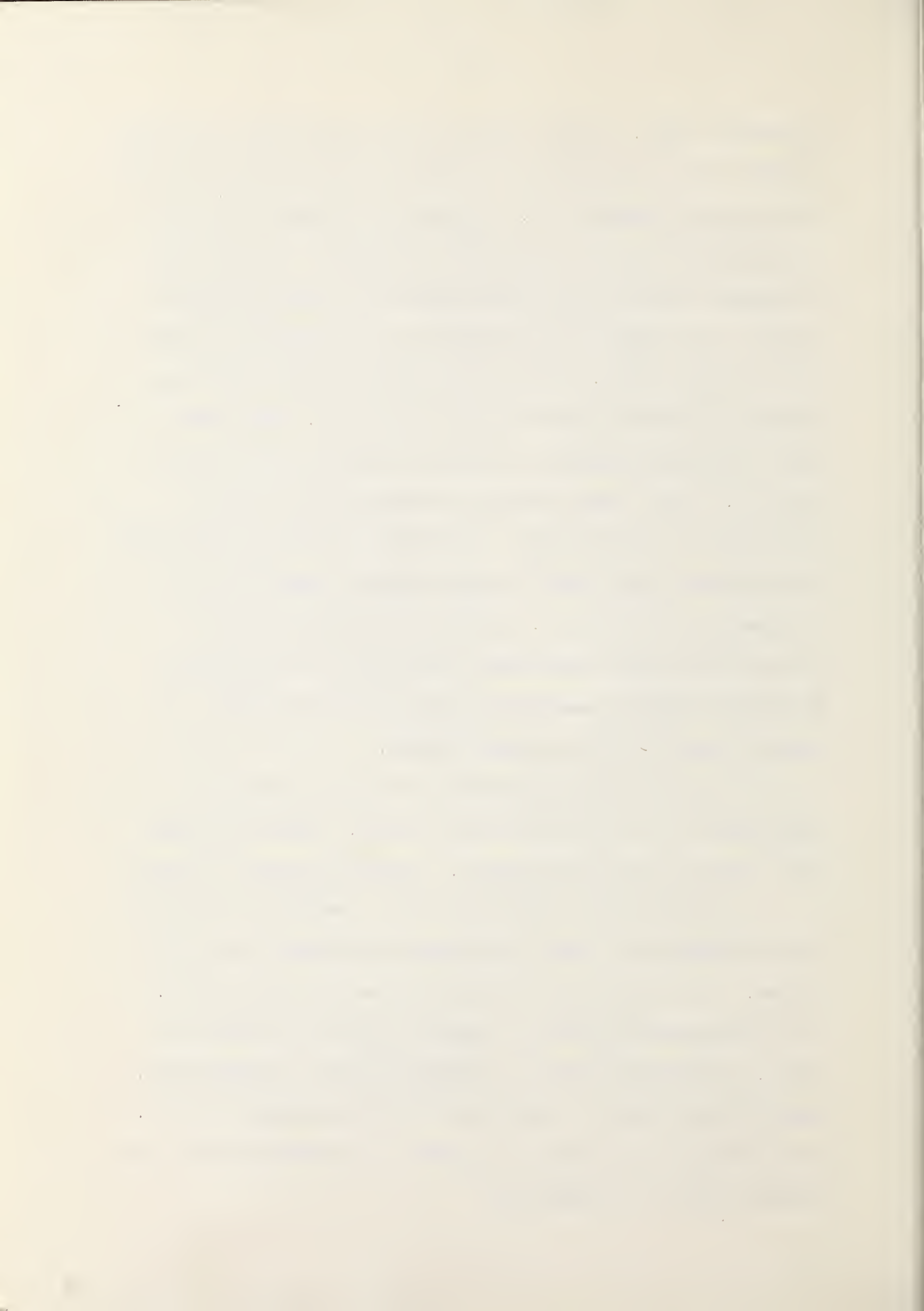
Heavy Mineral	Medonte M _H	Sebright M _H	Longford M _H	Coboconk	Medonte M _x
Opaque					
Pyrite	xxx	xxx	xxx	xxx	xxx
Magnetite	x				
Ilmenite	x				
Total Percentage Opaques	80	99	99	99	95
Non-opaque					
Apatite	xxx	xxx	xxx	xxx	xxx
Biotite	xxx	x	x	x	x
Epidote	x				y
Garnet	xxx	xx	xx	x	xxx
Hornblende	xxx	xxx	xxx	xxx	xxx
Kyanite	x			x	
Pyroxenes: Augite	x	x			
Acterine-Augite	x	xxx	xx	xx	xx
Diopside		xx	x	x	x
Hypersthene	xx	xx	xx	xx	y
Sphene				x	
Putile	x	x		x	x
Tourmaline	x	x	x	x	xx
Zircon Euhedra & Fragments	xxx	xxx	xxx	xxx	xxx
Distinctly Rounded	x	x	x	x	xxx
Total Percentage Non-opaques	20	1	1	1	5
xxx	Abundant (over 10%)	xx	Common	x	Rare to trace



Poldervaart (1956). Doubly terminated grains with simple and complex pyramidal faces are common, the stubbier varieties being more abundant than the slim, prismatic type. Some grains are broken so that one end of the grain only has a pyramidal termination. Inclusions vary in abundance and shape. Rod-shaped apatite(?) crystals, oriented parallel to the length of the crystal or with no particular orientation are fairly common. Also, dark irregularly shaped blotches and spherical gas and/or liquid inclusions are present. Some euhedra exhibit large solution-cavity notches or "nibbles" in the sides of the prism. Zoning, knee twinning, and overgrowths were noted in some grains but are not common. The sharply terminated crystals tend to be colorless, the slightly rounded varieties commonly darker due to abundant dusty inclusions. These two varieties make up to 85 per cent of the total zircon yield in the majority of the samples. The Medonte Mx zircon concentrate varies from this generality. It contains about 50 per cent euhedral zircons.

The broken angular fragments of crystals account for approximately 10 per cent of the total zircons. They are generally clear, but dusty grains were present. Conchoidal fracture is common.

The distinctly rounded grains form a minority in all samples except the Medonte Mx. Here they approach 50 per cent of the total zircons. In the other cases they vary from 5-10 per cent or less. The rounded grains are generally colored, pale yellow or very pale purple. Fissures and fractures are present as well as dusty grains. Inclusions are similar to those described for the euhedral crystals. Some zoning was also observed. No grains of the hyacinth variety were observed except in the Medonte Mx.



The low concentration of distinctly rounded grains suggests relatively little detrital contamination with the exception of the Medonte Mx sample.

Photomicrographs of representative zircons are shown in Plates 1, 2, 3, and 4.

Statistical Study

Statistical work was undertaken on the zircon concentrates of the Ontario bentonites in an effort to compare these zircons with those from known volcanic ash falls such as that described by Pitchie (1957). Poldervaart concluded from his study of zircons from various igneous rocks that, "Quantitative classifications of zircon concentrates are at all times to be preferred to qualitative classifications of zircons. Measurements of crystal size seem particularly helpful". Consequently, size measurements of individual crystals were carried out for, as Ritchie states, "the prime purpose of placing the study on a reproducible statistical basis".

Measurements and Results

Maximum length (L) and breadth (B) of individual crystals were measured with a micrometer eyepiece in a petrographic microscope. Broken crystals were excluded from the count. Elongation ($\frac{L}{B}$) was calculated for each grain and the three criteria plotted against frequency per cent. This resulted in three curves constructed on the basis of 200 measured crystals for each sample studied. For further details of the measurement and computation techniques, the reader is referred to the appendix.



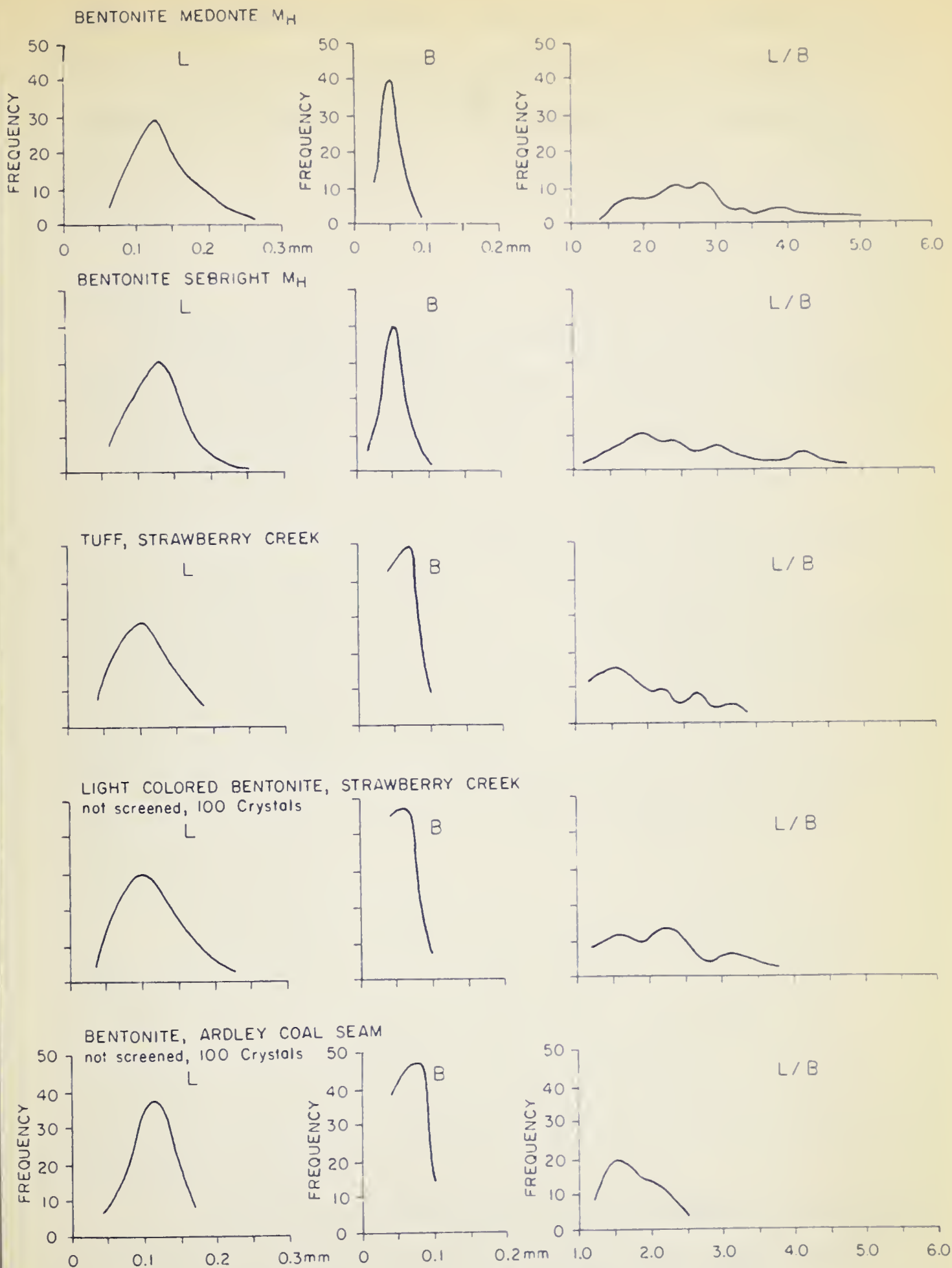
Statistical curves were constructed for two samples only, the Medonte M_H and the Sebright M_H . The Medonte M_x sample was considered to contain too great a proportion of detrital grains and the Longford and Coboconk samples did not contain a sufficiently large zircon crop to warrant analysing. Curves for the samples studied and those for comparison from the Kneehills Tuff (Upper Cretaceous) (after Ritchie, 1957) and the Ardley Coal Seam (Upper Cretaceous) (after Ritchie, 1957) are shown in Figure 4.

The length (L) curves for the zircons from the Ordovician bentonites show a greater proportion of longer crystals while the breadth (B) curves show a definitely greater proportion of slimmer crystals than those from Ritchie's Cretaceous Bentonites. Elongation curves are somewhat similar to those of Ritchie. Bimodal or trimodal effects are apparent in both cases, but the Ordovician zircons appear to have elongations spread over a wider range of values than do the Cretaceous zircons. A greater abundance of long, slim, zircon crystals is indicated for the Ordovician bentonites by the higher frequency percentage of elongations at greater than 3.5 units.

The length, breadth and elongation curves compare remarkably well for the Medonte M_H and Sebright M_H zircons. This factor suggests a contemporaneous ash fall and identical source for both locations.

Essential Components

The fraction of the bentonite "grit" sample remaining after the heavy minerals had been removed, was analysed for the type and abundance of essential components present. The fraction contained



ZIRCON SIZE - FREQUENCY CURVES

EACH SET OF CURVES IS BASED ON MEASUREMENTS OF 200 CRYSTALS
RECOVERED FROM GRADE SIZES LESS THAN 170 MESH BUT GREATER
THAN 270 MESH UNLESS OTHERWISE INDICATED

FIG. 4



quartz, feldspars, devitrified volcanic glass, clay aggregates, and carbonate grains. The feldspars and devitrified volcanic glass were given specific attention since they were used for potassium-argon age dating. The abundance of the essential components is given in Table 3.

Description

Feldspars - Several types of feldspars were noted in each sample, the most important being sanidine. The sanidine is not the most abundant feldspar as plagioclase is more common, but its presence give further indication of the volcanic origin of the bentonites. Sanidine occurs as generally clear, colorless to pale pink, angular, cleavage fragments. Some grains may show dark inclusions but conclusive evidence of transportation or decomposition is lacking. Sanidine made up from 20-25 per cent of the feldspar content of the samples. The Sebright sanidine grains appear to have more dark inclusions than were observed in the other samples. This mineral was distinguished from other feldspars by its small optic angle, less than 15° .

The remaining feldspars were grouped together as plagioclase, since twinning of any variety is almost completely absent. On the average, only two or three grains per slide showed either microcline or albite twinning. The plagioclase group is generally fresh looking and angular, however, dark inclusions and some alteration and etching is present.

The freshness of the feldspars varies somewhat from location to location. The Medonte M_H, Coboconk and Longford samples have a

large majority of clear angular grains while the Medonte Mx and Sebright samples show a greater proportion of etched and slightly altered grains. In the Medonte Mx case this feature correlates with the presence of abundant rounded zircons, rounded pyrite, and subrounded quartz to give more evidence of contamination.

Quartz - Occurs as angular, clear grains in most cases, the exception being the Medonte Mx sample which shows approximately 50 per cent rounded to subrounded grains. Dark inclusions may or may not be present but are not very common.

Devitrified Volcanic Glass - Occurs as light-to medium-brown grains with abundant dark inclusions. The darker brown color could be due to increased iron content. Variations in the intensity of brown also occurs between localities. The Medonte M_H sample has a greater abundance of the lighter brown grains than any of the other four samples. Between crossed nicols, the glass appears similar to a very fine grained volcanic rock fragment composed of an aggregate of grains with weak first order grey birefringence. A negative biaxial figure with a small optic angle was obtained from some of these fragments. This effect was probably due to a combination of the optical properties of each tiny individual grain. Possible relict glass structures (shards) were observed.

Clay Mineral Aggregates - These aggregates occur as very dark, dusty grains which are almost opaque except on thin edges. The birefringence shown around the edge of the grains is quite high and sparkly. The index of refraction appears to vary, at times being greater than 1.54 and in other instances less than 1.54.



Contaminants

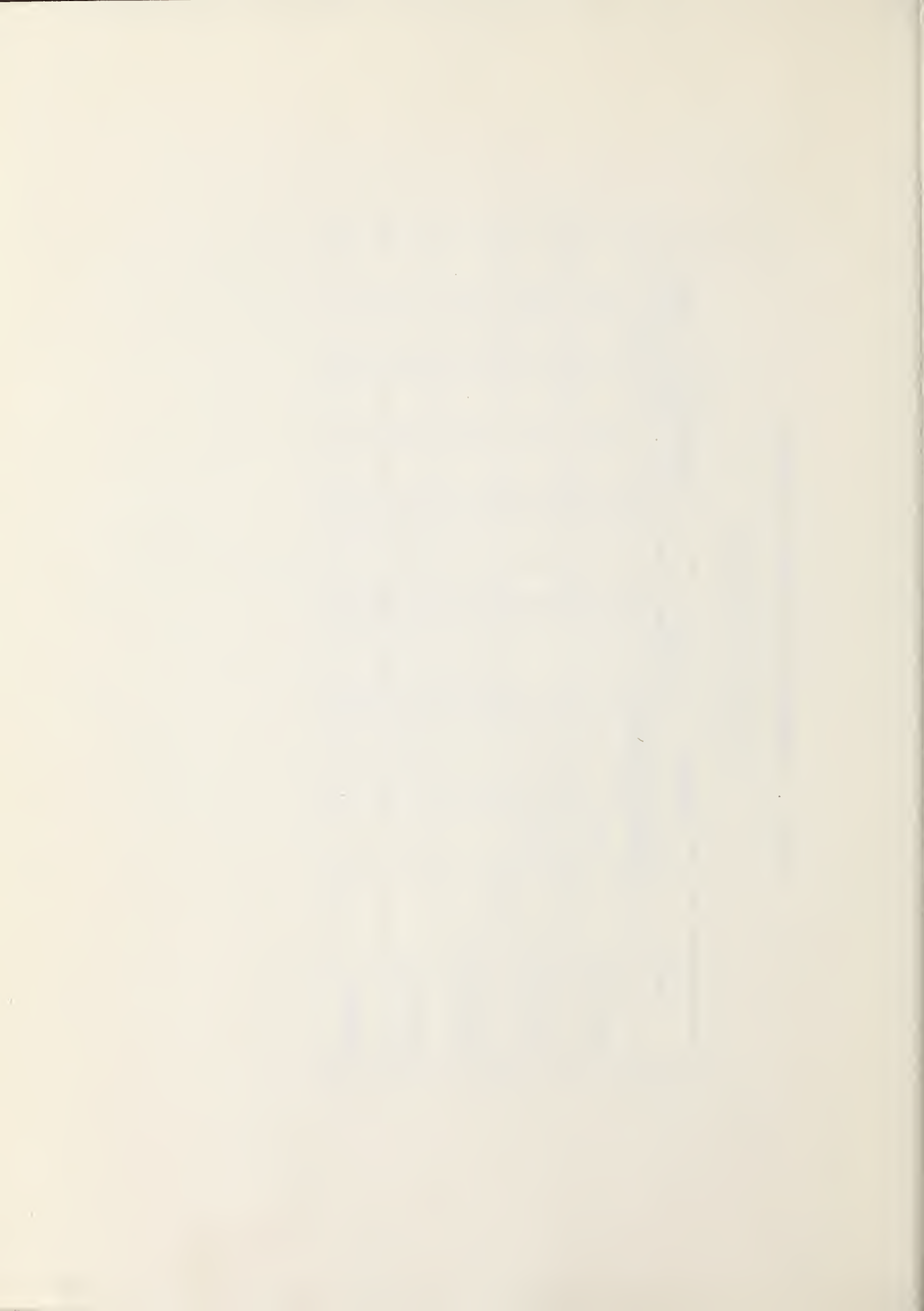
Carbonate Grains - The distinction between fine carbonate aggregates and clay aggregates is very difficult. Grains which seemed to have the very high calcite birefringence were called carbonates. Some subhedral single grains of calcite were also noted.

The dominant light mineral in all cases is a clay mineral, as would be expected in a bentonite. Quartz is not abundant except in the case of the contaminated Medonte Mx sample. Carbonate aggregates are present only in trace amounts. These grains could probably have been derived from the limestones above and below the bentonite. Feldspars are common in two samples only, the Medonte M_H and Medonte Mx. The higher concentration in the Medonte Mx sample could partly be accounted for by contamination, but in the Medonte M_H sample, the feldspars are definitely fresh and appear volcanic in origin. The abundance of devitrified volcanic glass also varies, being high in the Coboconk and Medonte M_H localities. There is a correspondingly lower percentage of clay aggregates in these two samples which suggests a less suitable environment for alteration of the glass to the clay minerals.

TABLE 3. PERCENTAGES OF ESSENTIAL COMPONENTS

(SILT AND SAND FRACTION)

Sample	Sanidine		Total Feldspar	Quartz	Devitrified Vol. Glass	Clay Aggregates
	%Felds	%Lights				
Medonte M _H	25	3	12	6	23	59
Sebright M _H	20	1	3	2	13	82
Longford M _H	-	-	3	1	12	84
Medonte Mx	27	4	14	20	13	53
Coboconk	-	-	2	2	24	72



CHAPTER FOUR

CLAY MINERALOGY

INTRODUCTION

The fraction of the bentonites less than 2 microns in size was subjected to x-ray diffraction tests and base exchange analysis in an attempt to make a positive identification. It was felt that a knowledge of the clay mineral constituents would aid in any provenance studies and also be a tool for comparisons with bentonites from other parts of North America and Europe.

X-RAY DIFFRACTION

Introduction

X-rays have been used for many years in the analysis of minerals. They are especially useful in clay mineralogy where the material is so fine grained that identification by optical properties is almost impossible. Two types of recordings may be attained by x-ray diffraction. The x-ray photo type is especially useful where accurate recording of large numbers of reflections is required. Unfortunately, the basal reflection of clay minerals, which is of prime importance in their identification, is often marred by shadows around the center of the photo. Thus, identification is made difficult. The x-ray diffraction pattern, however, is more useful since it is possible to determine the presence of mixed-layer clays as well as monomineralic types. Mixed-layer clays may then be analysed for the proportion of expanded to non-expanded layers present in the material. Both x-ray photos and patterns of the five bentonite samples are presented in this paper; the photos in Plate 9;

the x-ray patterns in Figure 6.

Preparation and Technique

Glass slides containing oriented flakes of the clay minerals were prepared for the x-ray diffraction in which a wide-range goniometer and a Brown recorder were used. Reflections from planes within the mineral were recorded as peaks on the pattern produced, (see Figure 6). To form the clay slides, approximately 5 gm. of the clay fraction of the bentonite were placed in 1000 ml. of water contained in a sedimentation cylinder, (Procedure for the isolation of the clay fraction is outlined in Chapter Three). It was found that by using this low concentration of clay, there was very little flocculation and any material coarser than 2 microns would settle out quite quickly. Consequently, the extensive time required for settling of normal sedimentation concentrations was shortened appreciably. The clay was allowed to settle for several minutes and then 100 ml. of the upper few inches of the suspension were pipetted off. The pipetted clay and water were drained into a 400 ml. beaker on the bottom of which was a glass plate ordinarily used for mineral mounting. The clay minerals were then allowed to settle out overnight in the beaker, after which time a thin coating of flat lying clay flakes had collected on the glass slide. The supernatant liquid was removed from the beaker with a pipette. A few hours at room temperature proved sufficient to dry the clay remaining on the slide.

The oriented clay mineral slides were x-rayed by Dr. L. Halferdahl of the Research Council of Alberta. He employed a Norelco x-ray diffractometer unit with a wide range goniometer, copper K α

radiation, and a nickel filter.

Standard settings of 35 kv and 20 mA were maintained on all runs to attain uniformity in the patterns. Each sample was x-rayed under three different conditions; untreated, glycolated, and heated. Glycolation was produced by immersing the samples in glycol vapors for 45 minutes at 50° C. The heated samples were maintained at 550° C. for 30 minutes.

The x-ray photos were produced on the x-ray diffractometer unit at the University of Alberta, Department of Geology. Small rods or spindles were made by mixing a minute quantity of the clay material with fingernail polish. The resulting mass was plastic at first and could be rolled into a very thin rod. The rod was then placed in the circular camera on the unit and x-rays shot through it. When a particle in the spindle had the correct orientation to fulfil the Bragg equation, x-rays were reflected onto the film in the camera where they were recorded. Only untreated samples were run on this x-ray unit.

Identification

Illite, montmorillonite, chlorite and vermiculite which are considered to be 2:1 clay minerals, have a similar basic structural unit. The type of cation which occurs between the units or layers determines the type of clay. For example, K^+ between the layers produces an illite; $(Mg,Al)(OH)_2^+$, chlorite; and Ca^{++} , Na^+ , Mg^{++} , and H_2O , montmorillonite or vermiculite. A simple monomineralic clay will contain the same type of cation between each layer, but if some units contain K^+ (illite) in the interlayer position, and others contain Ca^{++}

and H_2O (montmorillonite), a mixed-layer clay is the result. All possible combinations of the clay minerals have been found with up to four different clay types being present in one sample (Jonas and Brown, 1959).

Monomineralic clays produce a (00L) series of x-ray reflections which are an integral series of the (001) reflection. This is in turn a measure of the thickness of the unit cell, e.g. $10 \text{ \AA}$ for illite. Thus, d spacings giving the (00L) reflections for illite would be: (001) = $10 \text{ \AA}$, (002) = $5 \text{ \AA}$, (003) = $3.3 \text{ \AA}$, (004) = $2.5 \text{ \AA}$, (005) = $2.0 \text{ \AA}$, etc. The diffraction effects from a clay formed by two clay types are quite different from those produced by a single clay mineral. The (00L) values are average values resulting from the simultaneous scattering by both types of layers, i.e. instead of two (001) diffraction lines, one for each clay of the mixture, there is a single diffraction line located in an intermediate position. The location and intensity of the composite reflection will vary with the relative abundance of the different individual layers. For example, in a random mixed layer structure of $10 \text{ \AA}$ and $15.4 \text{ \AA}$, a strong reflection near $5 \text{ \AA}$ will occur from the combination of (002) of the $10 \text{ \AA}$ layers and (003) of the $15.4 \text{ \AA}$ layers. This reflection will tend to migrate toward the (003), $5.1 \text{ \AA}$ position as the proportion of $15.4 \text{ \AA}$ units becomes greater.

In the case of a mixture of clay minerals in which there is a regular interstratification of two types, a superstructure is built up having a larger unit cell. An integral series of (00L) reflections is obtained from this larger unit cell.

The theoretical basis for the identification of mixed-layer clay minerals has been developed in papers by Hendricks and Teller (1942), Bradley (1945, 1950, 1953), MacEwan (1949), and Brown and MacEwan (1950). For more detailed information regarding this phase of the study, the reader is referred to the works of the above men.

X-ray curves of the five bentonites studied are shown in Figure 6. Identification of the clay minerals present was largely made by comparison with the work done on mixed-layer clays by Weaver (1956).

He (Weaver) refers to the average reflections produced by nonexpanded $10 \text{ \AA}^0$ layers and expanded $17 \text{ \AA}^0$ layers as $(001)/(001)$, $(002)/(003)$ etc., indicating which two values combine to form the average value. This terminology will also be employed here. Figure 5 is a curve showing the migration of the $(001)/(001)$ peak as the percentage of expanded $17 \text{ \AA}^0$ layers is varied.

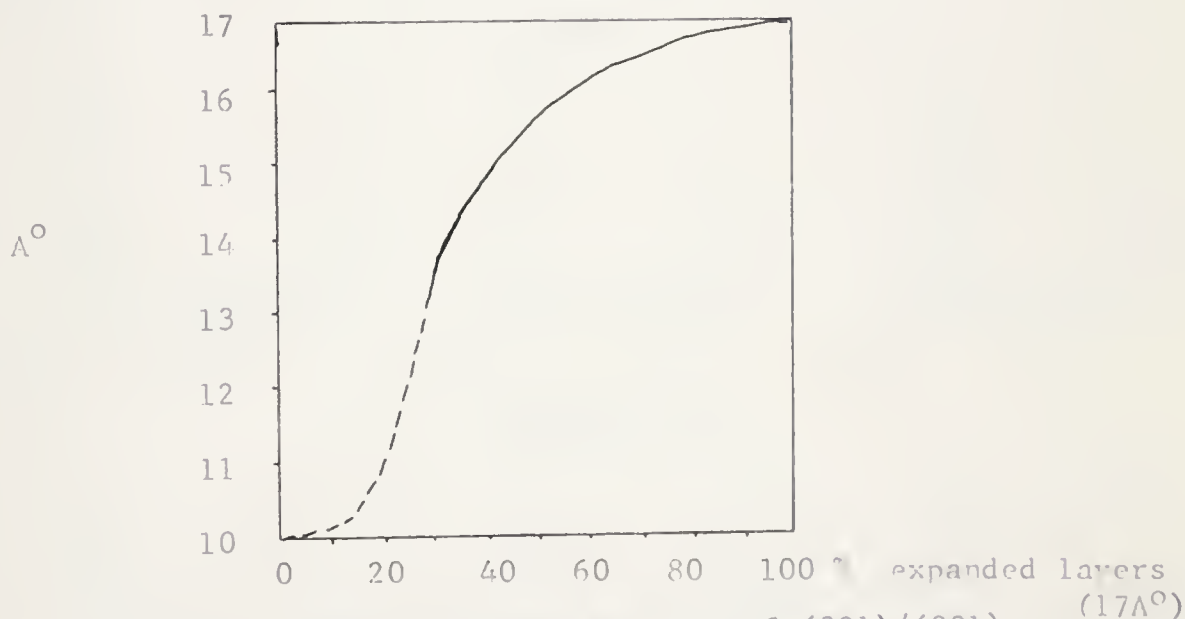


FIGURE 5. Curve showing migration of $(001)/(001)$ peak of randomly interstratified $10 \text{ \AA}^0$ and $17 \text{ \AA}^0$ layers. (after Weaver, 1956)

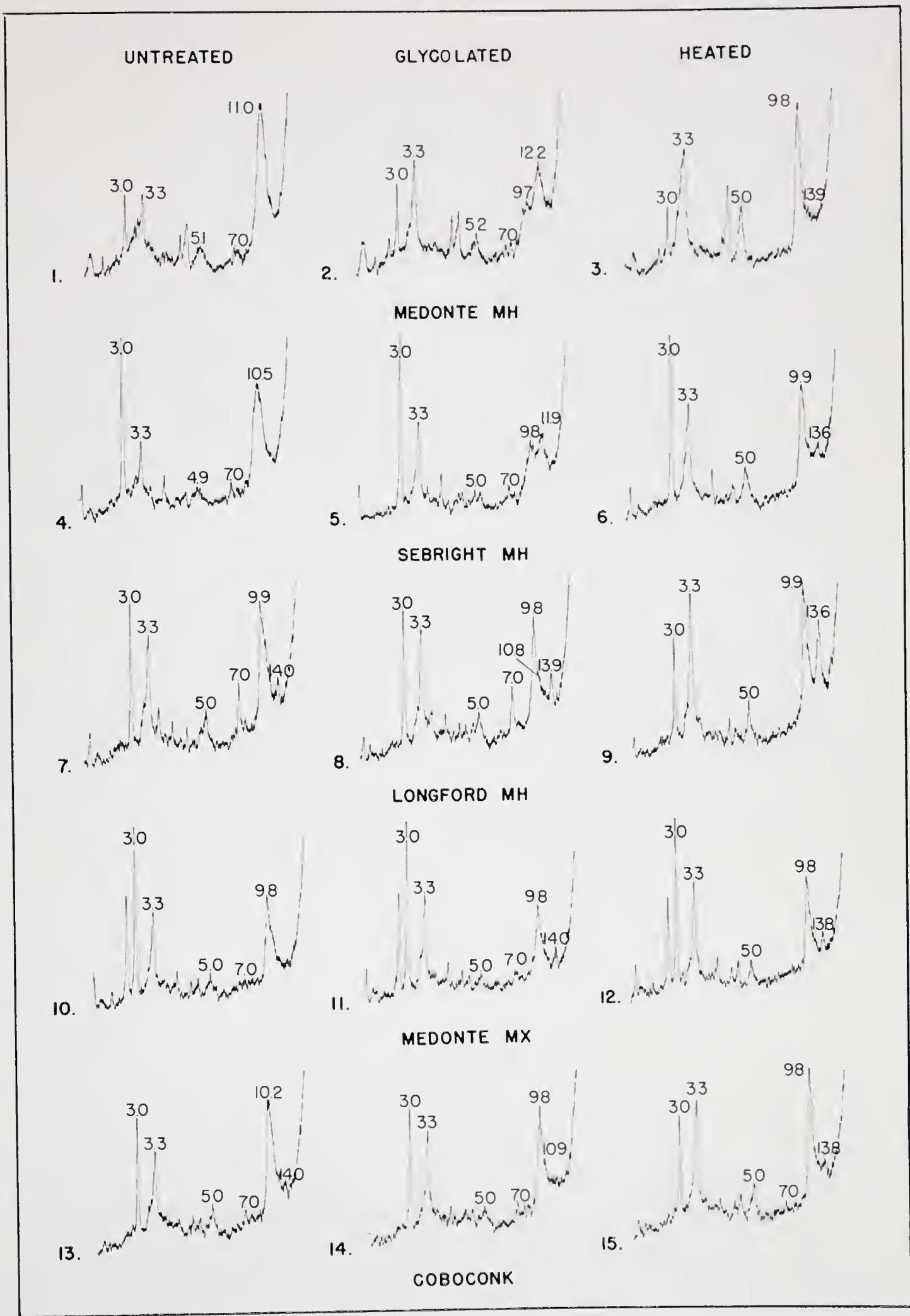


FIGURE 6 X-RAY DIFFRACTION CURVES



The Medonte M_H x-ray diffraction patterns (Figure 5; 1,2,3) show an 11 $\text{\AA}$ (001) peak for the untreated sample. This immediately indicates the presence of a mixed-layer clay with some expanded layers. Upon glycolation, the (001) peak splits into two observable maxima, one at approximately 9.7 $\text{\AA}$ and the other at 12.2 $\text{\AA}$. The 9.7 $\text{\AA}$ reflection is formed from a combination of energy from the (001) of the 10 $\text{\AA}$ layers present and the (002) of the 17 $\text{\AA}$ expanded layers. The 10 $\text{\AA}$ reflections combine with the 17 $\text{\AA}$ reflections to produce a (001)/(001) peak at 12.2 $\text{\AA}$. The (002)/(003) peak increases from 4.9 $\text{\AA}$ to 5.2 $\text{\AA}$ as the expanded material increases its unit thickness on glycolation. These facts indicate a mixed-layer aggregate containing from 25 to 30 per cent montmorillonite.

Upon heating the sample to 550° C., the expanded layers collapsed so that a (001)/(001) peak at 9.84 $\text{\AA}$ was produced. The heated clay also shows a small peak at 13.9 $\text{\AA}$ which indicates a minor amount of chlorite present. This is further substantiated by the fact that the 7 $\text{\AA}$ maxima((002) reflections) which are apparent in the patterns of the untreated and glycolated samples are completely obliterated from the curve for the heated sample. According to Weaver (1956) some OH^- ions are removed from the chlorite interlayer positions upon heating to 550° C. The second, third and fourth order reflections are destroyed and the curve contains only the first order peak which is shrunk to approximately 13.8 $\text{\AA}$ by the loss of OH^- .

The Sebright M_H clay (Figure 6; 4,5,6) contains from 20 to 25 per cent montmorillonite. This is indicated by the expansion of the (001)/(001) maxima from 10.5 $\text{\AA}$ to 11.9 $\text{\AA}$ upon glycolation. A



9.8 $\text{\AA}^0$ peak is formed from the (001)/(002) combination. Heating causes collapse of the expanded layers to a value of 9.9 $\text{\AA}^0$, and of the small amount of chlorite layers to 13.6 $\text{\AA}^0$. The (002) chlorite maxima is also destroyed after heating.

The Longford M_H clay, contains a considerable amount of chlorite, estimated at 20%. The (001) peak at approximately 14 $\text{\AA}^0$ can be clearly seen in the three curves in Figure 6; (7,8,9). A strong (002) chlorite maxima at 7.0 $\text{\AA}^0$ persists through glycolation but is practically destroyed on heating. There is very little montmorillonite present as evidenced by the (001)/(001) peak at less than 10 $\text{\AA}^0$. However, upon glycolation, a small maxima was produced at 10.8 $\text{\AA}^0$ and the (001)/(002) peak formed at 9.8 $\text{\AA}^0$. Heating contracted any expanded layers so that a 9.9 $\text{\AA}^0$ maxima resulted.

The x-ray patterns for the Medonte M_x clay (Figure 6; 9,10,11) indicate a dominance of illite with some chlorite and no montmorillonite. The (001) reflection is consistently less than 10 $\text{\AA}^0$ and does not increase after glycolation. The reason for a (001) peak at 9.8 $\text{\AA}^0$ is not entirely clear. Imperfections in the crystal lattice, however, could possibly reduce the cell thickness and thus give the smaller d spacing. Some chlorite is present as indicated by the 14.0 $\text{\AA}^0$ and 7 $\text{\AA}^0$ peaks. The 7 $\text{\AA}^0$ peak is destroyed upon heating.

The Coboconk x-ray curves are shown in Figure 6; (13,14,15). From 15 to 20 per cent montmorillonite layers are indicated here. The 10.2 $\text{\AA}^0$, (001)/(001) peak expands upon glycolation into a small peak at 10.9 $\text{\AA}^0$. The main maxima retracts to 9.8 $\text{\AA}^0$ as the (001)/(002)

reflections combine. The (003) $3.3 \text{ \AA}^0$ reflection and (005) $3.4 \text{ \AA}^0$ reflection nearly coincide producing a slight increase in intensity of the (003)/(005) peak. This increase is difficult to evaluate, however, since the clay reflection is marred by the presence of quartz reflections at the same location. Chlorite is also present in small amounts in this sample. A small $13.8 \text{ \AA}^0$ peak can be seen on the glycolated and heated curves. The $7 \text{ \AA}^0$ maxima are again obliterated on heating.

A summary of the clay mineral content of the different bentonite samples is given in Table 4.

SAMPLE	ILLITE	MONTMORILLONITE	CHLORITE
Medonte M _H	~ 70%	25-30%	Trace
Sebright M _H	~ 75%	20-25%	Trace
Longford M _H	~ 70%	< 10%	~ 20%
Medonte M _x	~ 90%	-	< 10%
Coboconk	~ 80%	15-20%	Trace

TABLE 4. Clay Mineral Content of Ontario Bentonites

In general, it may be said that the clay mineral constituents of the Ontario bentonites are fairly consistent throughout the samples studied. Slight variations in the illite:montmorillonite ratios of the mixed-layer aggregates are apparent, but in all cases there is a definite dominance of illite. The $14 \text{ \AA}^0$ mineral which occurs in minor amounts is believed to be chlorite. It apparently occurs as separate packets interspersed among, but not as part of, the illite-montmorillonite layers.

Weaver (1953) has found a similar clay mineral assemblage in some of his Pennsylvanian K-bentonites.

BASE EXCHANGE

Introduction

The atomic structures of clay minerals are such that they have the property of adsorbing certain cations and anions and retaining them in an exchangeable state. By treating these exchangeable ions with a solution of other ions, an exchange may take place. The adsorbed cations or anions are held around the outside of the clay particle so that the exchange reaction generally does not affect the structure of the silica-alumina lattice unit.

Since a greater quantity of information is available regarding cation exchanges, it was decided that a study of cation rather than anion exchange would give more useful information for determining the properties and identifying the clay fraction of the samples involved.

Base Exchange Capacity

The property of exchange capacity is measured in terms of milli-equivalents per gram or more frequently per 100 grams. However, there is no single capacity value that is characteristic of a given group of clay minerals. Consequently, a range in capacity must be considered for each group, since it may vary with many factors.

In general, montmorillonites and vermiculites (expanding type clay minerals) have the highest exchange capacities while illites and chlorites are intermediate. Kaolinite and halloysite clay minerals exhibit the lowest capacity values.

The relative concentration of cations in solution, respective

replacing powers of the different cations, degree of crystallinity of the clay mineral, and particle size play a large role in determining the exchange capacity of the clay.

Causes of Base Exchange

There are three major causes of exchange which vary in individual importance depending upon the type of clay mineral present.

1. Broken bonds around the edges of the silica-alumina units give rise to residual charges which may be balanced by the adsorbed cations. Thus, for clay minerals such as kaolinite and halloysite where this is the major cause of exchange, most of the adsorbed ions would be held on non-cleavage surfaces parallel to the C axis. The number of broken bonds will be increased if the particle size is decreased or if the lattice is distorted. This would increase the cation exchange capacity.

For illite and chlorite, broken bonds are an important cause for exchange, especially in well crystallized specimens. The remainder of the exchange is taken up by substitutions within the lattice. In montmorillonite and vermiculite the majority of substitution takes place within the lattice and only 20% (approximately) is due to broken bonds.

2. Substitution within the lattice.

Substitution of Al^{+3} for Si^{+4} in the tetrahedral sheet and of ions of lower valence, particularly Mg^{++} for Al^{+3} in the octahedral sheet results in excess charges which are frequently balanced by adsorption of other cations. In this case, the adsorbed ions are found on cleavage surfaces which in the clay minerals would be the basal cleavage.



The ions are held with different bonding strength depending on whether the initial substitution was in the octahedral or tetrahedral sheet. The charges resulting from substitution in the octahedral sheet would act through a greater distance than those resulting from substitution in the tetrahedral sheet and thus, cations attracted because of the former cause would not be held so strongly.

3. Hydrogen of an exposed hydroxyl may be replaced by a cation which would be exchangeable. This could occur around the broken edges of the clay minerals where some hydroxyls would be exposed. Thus, some exchange due to broken bonds could arise from replacement of H^+ of an exposed OH^- . In kaolinite and halloysite, the sheet of hydroxyls on one side of the basal cleavage plane would be an ideal place for exchange of this type to occur.

Fixation of Cations

Certain cations may be adsorbed by the clay minerals in a non-exchangeable or difficultly exchangeable state. K^+ fixation in illite is the most common example of this phenomena. The potassium fixation takes place by the emplacement of K^+ between the basal surfaces of the mineral in spaces between the oxygen atoms. The ionic radius of K^+ ($1.33 \text{ \AA}$) is such that it will fit very tightly into these spaces (diameter $2.77 \text{ \AA}$). It is this close fit plus the charge attraction that makes K^+ difficult to dislodge from the illite lattice. It has been found that "degraded" illites, i.e. those that are K-deficient may become rebuilt by the addition of K^+ .

Standford and Pierre (1946) discovered that the NH_4^+ ion can also be fixed by clays in a difficulty exchangeable form. NH_4^+ and K^+ are probably fixed by similar mechanisms.

PREPARATION OF MATERIAL

A portion of the clay fraction of the bentonite was mixed thoroughly by rolling and was then heated to 105° C. for one hour. No dispersing agent had been added at any time during the previous separation of the clay since every attempt was made to protect the samples from any reagent which might cause premature ion exchange.

ANALYTICAL METHODS

The method given by M.D. Forster (1951) was employed for the first cation analysis. Duplicate samples were run as checks and it was found that the results obtained were very ambiguous. Correspondence between duplicates was very poor and the total exchange capacity from the ammonium distillation was much lower than that obtained by summation of the individual exchanged ions. Consequently, a new method involving an equilibrium exchange was developed by Dr. H. Baadsgaard and the writer.

The Equilibrium Method of Ion Exchange

This new method was based on the Law of Mass Action. The assumption was that a high concentration of an easily adsorbed ion such as NH_4^+ , continually in contact with the clay material, would in time replace essentially all the exchangeable ions present. The procedure used was as follows:

To a 125 ml. glass tube, add 10 gm. of clay sample and 100 ml. of 1 N NH_4Cl . Seal the tube and after cooling, shake vigorously for approximately one minute. Repeat several times and then 4 to 5 times per day thereafter for two weeks. Store the tube in a place without wide temperature variations.

After allowing the clay to settle out completely (at least 24 hours), pipette out two 25 ml. portions of the clear supernatant liquid. In order to prevent any of the finest clay from entering the pipette aliquot a filter must be attached to the pipette tip (an effective filter may be made by compressing spun glass into a small piece of rubber or plastic tubing). Apply the even suction of a vacuum pump to draw off the aliquot. Determine the exchanged alkalies from one of the 25 ml. portions and exchanged calcium and magnesium from the other.

Calcium and Magnesium

Dilute the 25 ml. aliquot to approximately 100 ml. in a 250 ml. beaker, make slightly acid with HCl, heat to boiling and add 0.5 gm. $(\text{NH}_4)_2\text{C}_2\text{O}_4$ (in a filtered solution). Add NH_3 solution (50 per cent) dropwise with stirring to precipitate calcium oxalate. Allow the solution to stand overnight and then filter off the precipitate, washing with 1 per cent $(\text{NH}_4)_2\text{C}_2\text{O}_4$ solution. Save filtrate for magnesium determination. Dissolve calcium oxalate from the filter with a few ml. warm HCl (approximately 1:1) and wash the filter with 5 per cent HCl, then water. Reprecipitate Ca from the filtrate by adding approximately 0.1 gm. $(\text{NH}_4)_2\text{C}_2\text{O}_4$ to the acid solution, heat to boiling, and add 50 per cent NH_3 solution dropwise with stirring until methyl orange turns color (to yellow). Add 1 ml. NH_3 solution in excess and allow to stand four hours. Filter under suction on fritted glass filter (discard filtrate), washing first with 1 per cent ammonium oxalate solution and then with 3 portions of 5 per cent NH_3 solution. Wash the stem and back of the filter, rinse out the

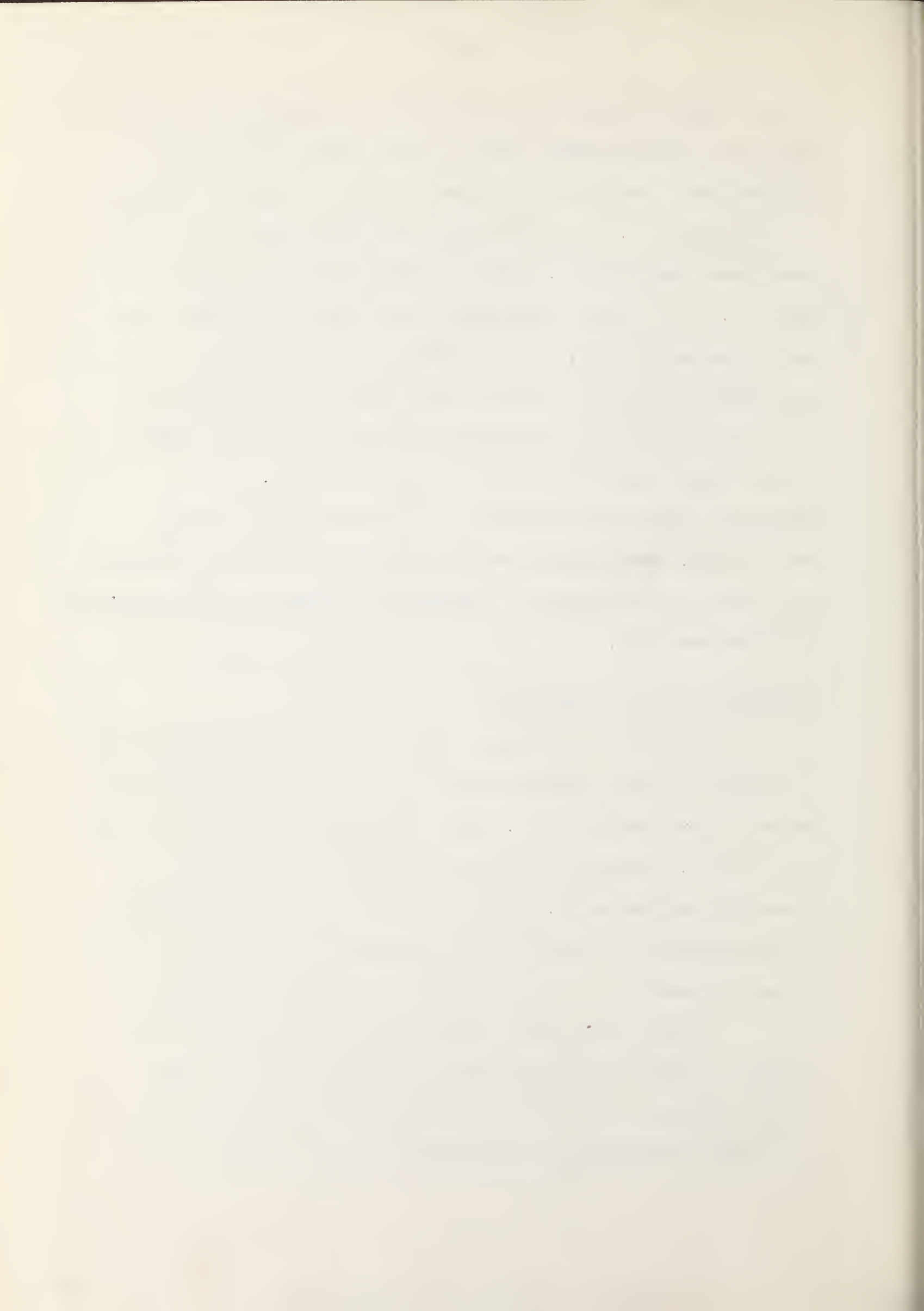
suction flask and dissolve the precipitate in a minimum amount of HCl. Rinse the filter thoroughly with 5 per cent H_2SO_4 and titrate the filtrate with standardized permanganate solution. Calculate meq. Ca^{++} .

Add 5 ml. 5 per cent HCl to the filtrate, heat, and to the warm (approximately 70°C.) filtrate (from the first Ca precipitation) add 5 ml. of 5 per cent 8-hydroxyquinoline solution (filtered). Then add approximately 15 ml. 50 per cent NH_3 solution and stir for approximately 5 minutes. Set the beaker aside to allow precipitate to settle and then filter off Mg-hydroxyquinolate through a weighed fritted glass crucible (heat for 1 hour at $100-105^\circ \text{C.}$, cool 40 minutes in a desiccator and then weigh), and wash with 2 per cent NH_3 solution. Heat crucible and dry precipitate in an oven at $100-105^\circ \text{C.}$ for 1 hour, cool 40 minutes in a desiccator and weigh as $\text{Mg}(\text{C}_9\text{H}_6\text{NO})_2 \cdot 2\text{H}_2\text{O}$. Calculate meq. Mg^{++} .

Alkalies (Sodium and Potassium)

Place the 25 ml. aliquot in a fused silica dish, evaporate to dryness and drive off the ammonium salts by increasing the temperature to approximately 450°C. Take up the residue in hot water, filter into a 50 ml. volumetric flask washing the dish and filter paper thoroughly with hot water. When cool dilute to the mark and employ a flame photometer to determine the exchanged Na^+ and K^+ , using comparable amounts of Ca and Mg in the flame photometer standards.

X-rays of the clay fractions showed the presence of other minerals, namely calcite and quartz, which have little or no base exchange capacity. The presence of quartz "dilutes" the original clay sample and lowers the exchange capacity, but some calcite could



dissolve and increase the amount of Ca^{++} determined as an exchanged ion. Attempts were made to determine the quartz content by the method described by Line and Aradine (1937), using fluorboric acid to extract the silicates present and determining the quartz from the insoluble residue. This method proved inadequate. Consequently, the quartz content was merely estimated. A value of 4 per cent was taken since it was found that the quartz content of the grit fraction was quite low (see Chapter Three) and this low value was thought to extend into the clay fraction. Further substantiating this assumption was the fact that the majority of the Ordovician meta-bentonites listed in the A.P.I. Research Project No. 49 and the Ordovician K-bentonite described by Weaver (1953) have a quartz content of from 5 per cent to a trace.

Carbon dioxide determinations on the clay fractions (see Table 5) revealed the presence of up to 17 per cent calcite. The neutral NH_4Cl -solution used in the base exchange will dissolve a certain amount of this calcite so that a correction for this additional calcium must be made. Assuming the neutral NH_4Cl -solution to effectively buffer the exchange medium at a $\text{pH} = 7.0$, knowing the total Ca^{++} concentration of the solution from the determination of calcium (see Table 7), and taking the HCO_3^- concentration as a measure of the calcite dissolved:



The Calcium from solution of CaCO_3 may be estimated using

$$\frac{\overline{\text{Ca}^{++}}}{\overline{\text{CO}_3^{--}}} = K_{\text{sp}} (\text{Calcite}) = 8.7 \times 10^{-9} \quad (25^\circ\text{C.}) \quad (1)$$

$$\frac{\overline{\text{CO}_3^{--}} \overline{\text{H}^+}}{\overline{\text{HCO}_3^-}} = K_2 (\text{carbonic acid}) = 4.4 \times 10^{-11} \quad (25^\circ\text{C.}) \quad (2)$$

Since for the case of the Medonte M_H sample, 3.69 meq. Ca^{++} / 10 gm. clay in 100 ml. 1 N NH_4Cl -solution were found (see Table 7),

$$[Ca^{++}] = 0.0185 \text{ M. From equation (1),}$$

$$[CO_3^{--}] = \frac{K_{sp}(\text{calcite})}{[Ca^{++}]} = \frac{8.7 \times 10^{-9}}{1.85 \times 10^{-2}} = 4.7 \times 10^{-7} \text{ M.}$$

Using equation (2) with this value for $[CO_3^{--}]$ and $[H^+] = 10^{-7}$,

$$[HCO_3^-] = \frac{[4.7 \times 10^{-7}][10^{-7}]}{4.4 \times 10^{-11}} = 1.07 \times 10^{-3} \text{ M} = [Ca^{++}]$$

In the 100 ml. of solution used in the base exchange on 10 gm. of clay sample there would then be

$$(2) (100) (1.07 \times 10^{-3}) = 0.214 \text{ meq. } Ca^{++}.$$

derived from the solution of some of the calcite in the clay sample. For a 100 gm. clay sample, approximately 2 meq. of excess calcium would be introduced. The Ca^{++} values in Tables 6 and 7 were corrected using this value.

Sample	Sample Weight (gm)	Volume CO_2 (ml)	% CO_2
Medonte M_H	0.2500	3.75	2.8
Sebright M_H	0.1502	13.24	16.6
Longford M_H	0.2502	16.94	12.8
Medonte M_x	0.2501	21.41	16.2
Coboconk	0.2501	14.49	11.0

TABLE 5. Carbon Dioxide Content of the Clay Fractions



Results

Duplicate pairs were run for each bentonite sample analysed so that a check on the values obtained could be made. Data procured on the exchanged cations by the Equilibrium Method checks between the duplicates quite well for the most part. The few apparent differences could be due to analytical technique rather than any variation in the clay sample or inadequacies of the method. However, the generally good correspondence between duplicates makes the results appear quite reliable (Table 6).

Low values are given for Na^+ and K^+ for sample #2 of the duplicates (Medonte M_H) since the leachate aliquot containing those ions was spilled and some solution lost. The values for both Na^+ and K^+ are proportionately lower, however, which substantiates reliability claims for that sample. There is no Ca^{++} value for sample #9 (Longford M_H) because of the lack of a definite end point in the permanganate titration. In both the above cases total exchange capacity was calculated on the basis of the one unspoiled sample of the pair. Final corrected and averaged values for the exchanged ions; Ca^{++} , Mg^{++} , K^+ , and Na^+ , and for the total base exchange capacity are given in Table 7.

Interpretation

X-ray diffraction data on the bentonite samples from Ontario indicate that the clay fraction is composed of a mixed-layer aggregate with illite predominant over montmorillonite. Small chlorite packets were also noted. (For determination by exchange capacity of the amount of montmorillonite present in the clay, chlorite may be grouped with illite since their exchange capacities are similar.



TABLE 6.
BASE EXCHANGE DATA SHOWING CORRESPONDENCE BETWEEN DUPLICATE SAMPLES
FOR SOME ORDOVICIAN BENTONITES FROM ONTARIO

Sample	Sample Weight (gms)	Milli-equivalents/100 gm. sample (dry basis, 110°C.), Corrected for quartz and CaCO ₃				
		Ca ⁺⁺	Mg ⁺⁺	K ⁺	Na ⁺	B.E.C.Sum
No. 1 Medonte M _H	10.0000	36.3	8.9	1.9	0.7	47.8
No. 2 Medonte M _H	10.0001	36.2	8.6	(1.4)*	(0.5)*	46.7
No. 3 Coboconk	10.0001	20.4	0.8	0.7	0.5	22.4
No. 4 Coboconk	10.0001	20.9	1.4	0.7	0.5	23.5
No. 5 Medonte M _x	10.0000	15.7	4.2	0.9	0.8	21.6
No. 6 Medonte M _x	10.0000	15.8	4.0	0.9	0.6	21.3
No. 7 Sebright M _H	10.0000	24.2	1.7	1.1	1.0	28.0
No. 8 Sebright M _H	10.0003	23.8	1.5	1.1	1.0	27.4
#No. 9 Longford M _H	10.0003	-	1.1	1.1	0.6	-
No. 10 Longford M _H	10.0000	28.7	1.4	1.1	0.7	31.9

*Sample spilled. ‡ No end point with permanganate titration.

Thus, only illite:montmorillonite ratios will be given with the understanding that a minor amount of the calculated illite will actually be chlorite). The base exchange capacities determined in this analysis also indicate a dominance of illite.

The term illite should be used with care, however, since it was originally proposed by Grim, Bray and Bradley in 1937"as a general term, not as a specific clay mineral name for the mica-like clay minerals." (Grim, 1953). Yoder and Eugster (1955), conclude that illite should be used only as a field term, most illites actually being various polymorphs of mica. They also state that "Proof has yet to be obtained that the materials now called illites are not all mixed-layer structures." In this paper the term illite will be employed to represent a mica-type clay mineral, composed of very small particles (of the order of 1 to 2 microns or less), with a $10 \text{ \AA}^0$ C-axis spacing and a low base exchange capacity. This general definition was employed to facilitate comparisons with previous work and also since the complete analysis required to determine exactly which polymorph of mica was present could not be undertaken.

The low base exchange values indicate a predominance of illite in the clay according to published data on mixed-layer aggregates (Ormsby and Sand, 1954) (Byström, 1956). Comparison of data with that of these workers is shown in Table 8.

Illite:montmorillonite ratios were determined from a straight line graph prepared by Ormsby and Sand (1954) in which base exchange capacities had been plotted on the ordinate and percentage montmorillonite on the abscissa.

TABLE 7. BASE EXCHANGE CAPACITIES OF BENTONITES FROM ONTARIO

Milli-equivalents/100 gm. sample (Dry basis, 110° C), average values											
Sample	Uncorrected for Quartz					Corrected for Quartz					B.E.C. Sum
	Ca ⁺⁺	Ca ⁺⁺ Corrected for CaCO ₃	Mg ⁺⁺	K ⁺	Na ⁺	Ca ⁺⁺	Mg ⁺⁺	K ⁺	Na ⁺		
Medonte M _H	36.9	34.9	8.5	1.8	0.7	36.3	8.8	1.9	0.7	48	
Sebright M _H	25.1	23.1	1.5	1.1	1.0	24.0	1.6	1.1	1.0	28	
Longford M _H	29.6	27.6	1.2	1.1	0.7	28.7	1.3	1.1	0.7	32	
Medonte M _x	17.2	15.2	3.9	0.9	0.7	15.8	4.1	0.9	0.7	21	
Cobocouk	21.9	19.9	1.1	0.7	0.5	20.7	1.1	0.7	0.5	23	

Table 8 shows a good correspondence between illite:montmorillonite ratios determined from base exchange capacities, and those determined from x-ray, chemical analysis and D.T.A. data. This close similarity suggests that the base exchange capacities determined for the Ontario bentonites are reliable and that base exchange may be used as an auxiliary analytical tool for evaluating mixed-layer aggregates.

It is worthy of note that the bentonites from eastern United States, (Weaver's and Bradley's samples) have similar base exchange capacities and illite:montmorillonite ratios to the bentonites from Ontario. Byström's Type II bentonites also compare favourably with the Ontario samples but her Type I bentonites are quite different. These latter samples can not be compared rigorously with the eastern North America bentonites since they have been taken from a much thicker bed. Byström points out that the thinner beds (Type II) may have been more accessible to the potassium of the sea water and were thus altered to a greater extent from montmorillonite to illite.

Another interesting point is the high K_2O content of the Ordovician bentonites; the exception to this again being the Type II bentonites from Sweden. This factor further substantiates the claim of a high illite percentage in the clay fraction. Table 8 illustrates (in columns 1, 2, and 3) the low percentage of potassium actually exchanged. K^+ fixation which is common in illite could account for this feature.

The A.P.J. mixed-layer aggregates have not had extensive analysis completed on them, so that it is not surprising that the data does not check too well. However, the one Ordovician sample,

TABLE 8. COMPARISON OF BASE EXCHANGE CAPACITIES FOR A VARIETY OF BENTONITES

Sample	Exchangeable K ⁺ meq/100 gm.	%K ₂ O	K ⁺ meq/100 gm.	B.E.C. meq/100 gm.	Ratio Illite:Mont. From B.E.C.	Ratio Illite:Mont. From x-ray D.T.A. & Chem. Analysis
Medonte Mh	1.9	5.08	106	48	58:42	70:30*
Sebright Mh	1.1			28	82:18	80:20*
Longford Mh	1.1			32	80:20	90:10*
Medonte Mx	0.9			21	90:10	100:0 *
Coboconk	0.7			23	87:13	85:15*
Byström's Data						
Type II Bentonite No. 1	2.2	5.3	110	46	59:41	3:2
Type II Bentonite No. 2	1.9	-	-	46	59:41	3:2
Type II Bentonite No. 5	3.0	4.3	90	49	57:43	3:2
Type II Bentonite No. 6	2.6	4.8	100	53	50:50	3:2
Type I Bentonite No. 4	2.2	2.5	52	66	48:62	1:4
Type I Bentonite No. 7	4.2	2.9	61	65	49:61	1:4
Ormsby and Sand's Data						
Metabentonite (Weaver)		5.89	122	27.2	84:16	3:1 or 4:1
Mixed-layer aggregate Bradley				43.0	54:36	1:1
M.L.A. A.P.I. #31				80.5	18:82	Illite dominant
M.L.A. A.P.I. #32				83.9	14:86	1:1
M.L.A. A.P.I. #41		4.4	91	30.0	80:20	Illite dominant

* X-ray only.

A.P.I. No. 41 from Tazewell, Virginia has a high K_2O content, similar base exchange capacity and similar illite:montmorillonite ratio to the other North American Ordovician bentonites.

Variations in base exchange capacity within the same M_H horizon of the Medonte, Sebright and Longford samples can be directly related to the type and abundance of the clay minerals present. The Medonte M_H has a relatively high base exchange capacity and a correspondingly high percentage of montmorillonite, while the Sebright and Longford samples have a lower percentage of montmorillonite and thus, a lower base exchange capacity.

The Coboconk and Medonte Mx bentonites represent two horizons which differ stratigraphically from the M_H ; the Coboconk lying above and the Mx below. These two samples are significant in that they show base exchange capacities lower than any of the M_H samples. However, being part of different ash falls one would hardly expect these beds to have identical properties to the M_H samples. Possibly, differences in the composition of the original ash and/or variations in the environment of deposition have caused the slightly higher ratio of illite to montmorillonite.

CHAPTER FIVE

GEOCHRONOLOGY

Introduction

Age dating was undertaken to establish an absolute age for the Middle Ordovician. There has been some controversy over the validity of the ages on the Holmes time scale for Lower Paleozoic rocks. Various workers, Kulp (1959) and Mayne, et al (1959), have determined, indirectly, consistently older dates for these strata. It was felt that the Ontario bentonites would provide an excellent opportunity to derive a direct absolute age for Ordovician sediments with an established paleontological position and thus aid in straightening out the controversy. The presence of sanidine and a devitrified volcanic glass material suggested the employment of the potassium-argon method.

Preparation of Material

The isolation of feldspars for the potassium-argon age determination is described in detail in Chapter Three. It is sufficient to say here that the use of heavy liquids (tetrabromoethane and acetone) and the Frantz separator were fairly efficient in removing extraneous quartz, clay aggregates and carbonate rock fragments. Statistical counts on Canada Balsam mounts of the samples dated indicated that a considerable proportion of other material still remained in the sample. The dominant reasons for this were the small initial volume of bentonite and the low percentage of feldspars in the "grit fraction." Repeated fractionations to

acquire a more purified product would have resulted in too small a sample of feldspars to be analysed accurately. Approximately 50 per cent of the total age dated feldspars for both the Medonte M_H and Sebright M_H samples were determined to be sanidine. The remainder of the feldspars were plagioclase, some of which may have been detrital.

The Medonte M_H devitrified volcanic glass was quite common throughout the grit fraction. Consequently, a 75 per cent pure sample was obtained.

The Medonte M_H feldspar and volcanic glass samples were analysed for per cent K₂O by the J. Lawrence Smith method. The per cent K₂O in the Sebright M_H feldspars was determined by flame photometry on a Perkin Elmer flame photometer. H. Baadsgaard extracted the argon from the samples with the argon train at the University of Alberta. Mass spectrometer measurements were done on the mass spectrometer in the Physics Department, University of Alberta.

Results

The potassium-argon age determinations calculated by H. Baadsgaard and the writer are as follows:

<u>Run No.</u>	<u>AK No.</u>	<u>Sample</u>	<u>Material Dated</u>	<u>Potassium Analysis</u>	<u>A⁴⁰/K⁴⁰</u>	<u>Age</u>
62	58	Medonte M _H	Feldspars	6.12% K	0.0459	654 m.y.
68	62	Sebright M _H	Feldspars	3.48% K	0.0460 ₆	656 m.y.
66	57	Medonte M _H	Devitrified Volcanic glass	5.13% K	0.0163 ₅	260 m.y.

The dates for the above samples were calculated using:

$$\begin{aligned} \lambda_e &= 0.583 \times 10^{-10} \text{ yr}^{-1} \\ \lambda_\beta &= 4.89 \times 10^{-10} \text{ yr}^{-1} \\ R &= 0.119 \end{aligned}$$

Interpretation

The three ages of 656, 654 and 260 m.y. are purely tentative at this time. A high air contamination factor in the argon from the devitrified volcanic glass, and very poor mass spectrometer runs for all three samples make any interpretation of these values meaningless. A portion of the extracted argon from each sample has been retained and will be run at a future date. It is hoped that less adverse conditions will be encountered at that time.

It may be said, however, that the feldspar ages of 656 and 654 m.y. suggest contamination by detrital grains. The very low age (260 m.y.) obtained from the volcanic glass may indicate argon leakage from this material.

Zircon from a bentonite bed in the Middle Ordovician (Trenton) Martinsburg shale at Fisher's Hill, Virginia, yielded a Pb/ age of 353 m.y. (Carroll, 1959). This age is almost within the range obtained for igneous rocks of Ordovician age, 360-440 m.y., on Holmes' time scale (1947).

A sample of biotite from the Upper Ordovician bentonites of Kinnekulle, Sweden, was forwarded to the Department of Geology at the University of Alberta by Miss A. Byström. Potassium-argon dating of the biotite revealed an age of 410 m.y. This date agrees favourably with that of Holmes for the Upper Ordovician (Caradocian).

CHAPTER SIX

PROVENANCE AND ENVIRONMENT OF DEPOSITION

Employing the data presented previously, evaluation may be made regarding the probable nature of the source material, the area of its origin, the manner of its deposition and the mode of formation of the clay minerals.

Source Material

The presence of many of the features characteristic of altered volcanic materials (Ross, 1928) indicates that the bentonites are volcanic in origin. These features include euhedral and zoned zircons, euhedral apatite, a material believed to be devitrified volcanic glass, and a feldspar with a small optic angle (sanidine). The heavy and light minerals may all occur in volcanic rocks. (Kyanite and microcline are exceptions and are obviously detrital contaminants.) The presence of an illite-montmorillonite mixed-layer clay mineral is also apparently an indication of an altered volcanic material, similar clay aggregates having been found in the Ordovician bentonites of the eastern United States and in Sweden. The abrupt lithologic change from a limestone sequence into a thin zone of clay material is another feature which can be best explained by a volcanic ash fall.

Identification of the composition of the source material is made difficult by the alterations which have taken place. However, the sparsity of quartz phenocrysts, the presence of diopsidic augite, aegirine-augite, hornblende, biotite, and a large percentage of potash

feldspar indicate a source material of trachytic or more likely latitic composition (Williams, Turner and Gilbert, 1955). This conclusion is similar to the one drawn by Weaver (1953) for the source rock composition of his Pennsylvanian K-bentonites. The mineral assemblages are quite similar in both cases although the Ontario bentonites have a greater variety of heavy minerals present. The Ordovician bentonites from Kinnekulle, Sweden, described by Bystrom (1956), have a sparse mineral assemblage and a greater percentage of quartz. Consequently, a dacite or rhyolite type source material has been postulated for them.

Source Area

Bentonites of similar age and characteristics to the Ontario bentonites occur over a wide area of the eastern United States (see Chapter Two). It is postulated that a very active period of volcanic activity occurred at this time which produced a number of ash falls. Some of the volcanic material may be traced as far as Minnesota to the west, New York state to the east, and south to Alabama (Ross, 1955). The ash falls become coarser to the southwest (Ross, 1955) and also thicker to the south (Kay, 1931). This would indicate a volcanic vent somewhere in the southern states. Kay (1935) states "... the relatively great thickness of altered volcanic rocks in southwestern Virginia suggests that an active center of vulcanism was in the adjacent crystalline region of North Carolina" It seems distinctly possible that this region could have been the source area for the Ontario bentonites. Although this

would involve hundreds of miles of ash transport, the continuity of the volcanic material from Virginia to Ontario indicates a similar source. Information regarding recent volcanic explosions provides evidence for fine volcanic dust encircling the world (Holmes, 1946). Thus, in view of the great volume of pyroclastics ejected, it does not seem unreasonable that some part of it was carried as far north as the Lake Simcoe region.

Depositional Environment

The wide lateral extent and the nature of the associated rocks, which are mainly limestone, indicate that the original volcanic material was laid down in shallow seas saturated with CaCO_3 . The fossil assemblage also points to marine deposition. The presence of limestone beds above and below the bentonite horizons indicates a pH (acidity-alkalinity) of at least 7.8, and a low landmass around the sea. Very little clastic material was being transported into the depositional basin as evidenced by the low detrital mineral content of the bentonites. The abundance of authigenic (?) pyrite can only be explained by reducing conditions near or below the depositional interface. Krumbein and Garrels (1952) believe that the Eh (oxidation-reduction potential) may be lowered and more favourable to the formation of pyrite below the depositional interface. This appears to be the case here since the high pH required to precipitate CaCO_3 would probably tend to restrict the reduction of Fe^{+3} to Fe^{+2} in the sea water.

Formation of the Clay Minerals

The clay minerals are presumably the major alteration product of the original volcanic material. However, as Ross and Hendricks (1945) state, "... the conditions under which alteration to bentonite takes place are not fully known and probably differ for different deposits...." These workers suggest that bentonites high in potassium were formed by alteration of tuffs in sea water while bentonites composed almost entirely of montmorillonite were formed by circulating ground waters. Weaver (1953) postulates that alteration of volcanic glass to form the Pennsylvanian K-bentonites occurred in sea water.

It is possible that the decomposition of the original volcanic glass of the Ontario bentonites could also have taken place in the sea. However, it seems more likely that calcium clay minerals (montmorillonite) would be produced due to the high concentration of calcium ions in the sea at that time. Leaching of the source material by the marine waters would remove potassium and probably some silica but would not extract any calcium. Thus, the clay composition would remain as montmorillonite as long as the calcium-saturated sea was in contact with the bentonite. Subsequent leaching by ground waters with a high potassium content could, at a later date, remove most of the calcium from the clay and substitute potassium in its place. This phenomenon would have to occur in at least 70 per cent of the layers to produce the random mixed-layer structures observed.

The reason for the presence of small packets of chlorite

dispersed among the mixed-layer aggregates is not entirely clear. Chlorite is trioctahedral compared to the dioctahedral illites and montmorillonites and has an additional brucite layer in its structure. No evidence has been found to indicate whether the formation of this mineral was by the action of ground water solutions or by the action of the sea water. Whichever the case may be, an abundance of magnesium ions must have been present in the altering solutions.



The presence of limestone beds above and below the bentonites and a marine fossil assemblage in the limestone indicate deposition of the ash in a shallow sea with a pH of at least 7.8. Postulation of reducing conditions near or below the depositional interface is verified by the abundant authigenic pyrite contained in the "grit" fraction.

Potassium-argon age dating of feldspars and devitrified volcanic glass fragments yielded ages of 656, 654 and 260 m.y. It is felt that these figures should be regarded as tentative only, since high air contamination and poor mass spectrometer recordings render any interpretations invalid. The remaining extracted argon samples will be run at a future date.

The extremely old feldspar ages of 656 and 654 m.y. suggest contamination by detrital grains while the young age of 260 m.y. for the volcanic glass indicates possible argon leakage.

Ages of 353 and 410 m.y. have been determined for Ordovician bentonites from Virginia (Carroll, 1959) and Sweden respectively.

CHAPTER SEVEN

CONCLUSIONS

Heavy mineral assemblages and the presence of devitrified volcanic glass and a high temperature feldspar point towards a source rock for the Ontario bentonites which is volcanic in origin. The composition was probably that of a latite.

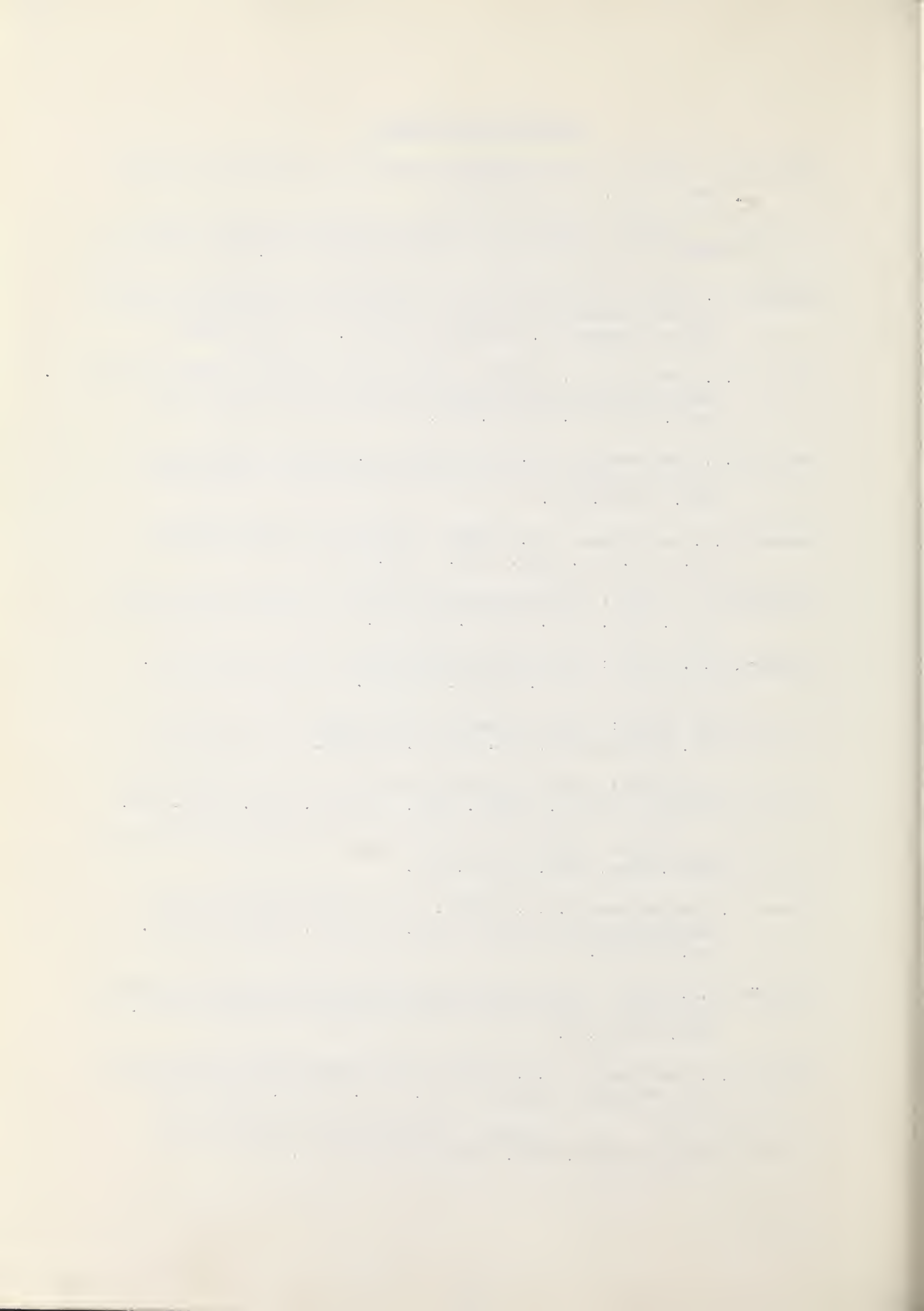
Base exchange and x-ray analysis on the clay fraction indicate a mixed-layer aggregate clay material which is thought to have altered from the original volcanic ash. Illite always predominates over montmorillonite in the mixed-layer clay, and some small chlorite packets have been determined interspersed among, but not as part of, the predominant, interstratified layers.

Close similarities to bentonites in Kinnekulle, Sweden, and Pennsylvania are apparent. The Ontario bentonites can be correlated locally and with those of the eastern United States and it is postulated that the whole region from Virginia to Ontario was the recipient of similar ash falls. Increasing thickness and coarseness of the bentonites toward the south indicate a possible source area in the crystalline region of North Carolina.

Alteration of the volcanic ash to the characteristic mixed-layer clays may have taken place in two stages. First; the calcium saturated Ordovician seas leached out potassium and some silica. Calcium was probably added to the residual structure forming a montmorillonite type clay mineral. The second stage occurred at a later date when ground waters with a high potassium content exchanged potassium for calcium in approximately 70 per cent of the layers. A mixed layer clay with a large proportion of illite was the result.

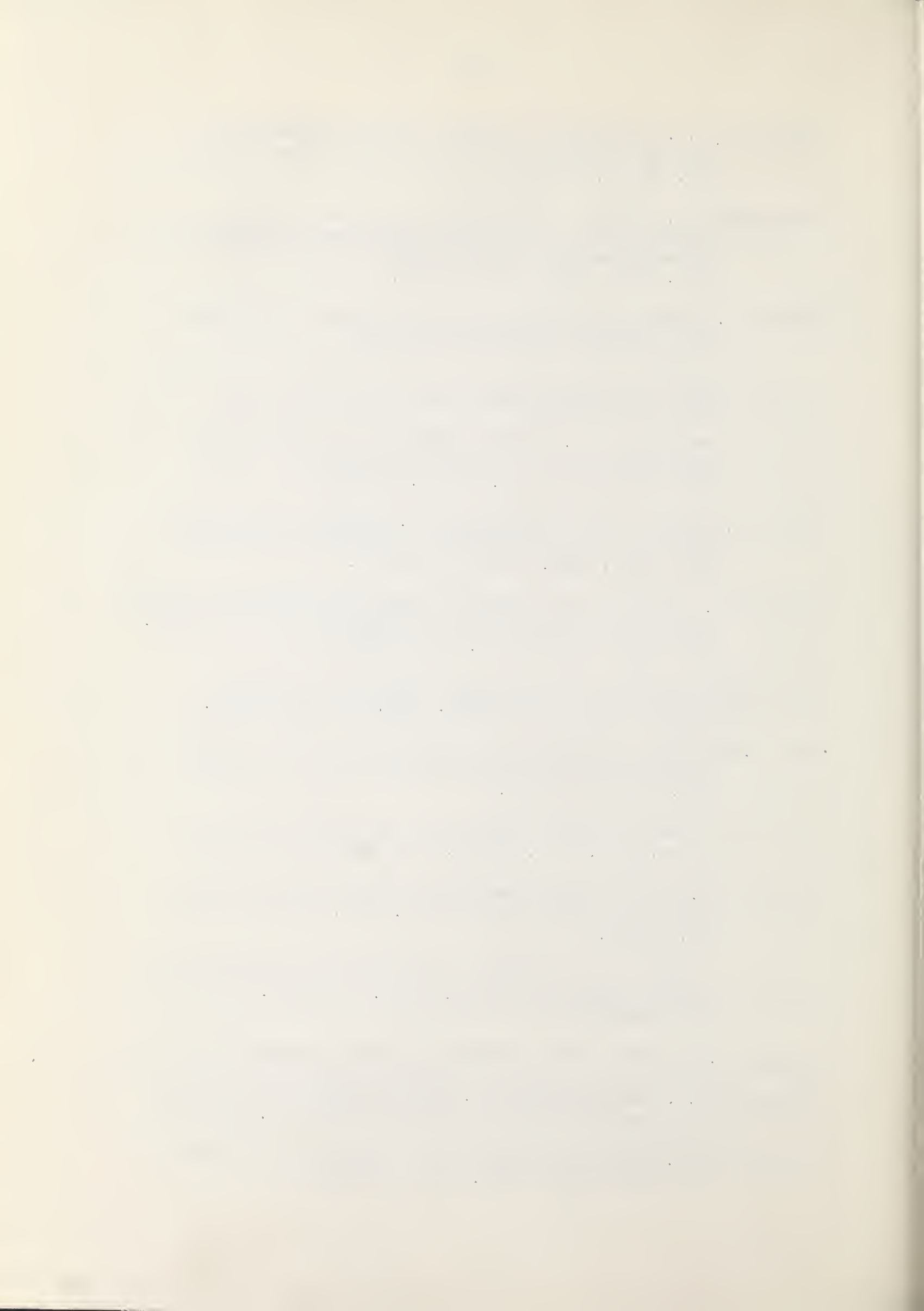
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APPENDIX A

Method of Preparing Size-Frequency Curves

The method described here is similar to the one developed by Smithson (1939). However, a variation in the procedure for determining the frequency of grains in the different size ranges is presented. The type of elongation diagram is also different from that of Smithson. It is worthy of note that other workers (Poldervaart, 1956, Ritchie, 1957) have used this method before but the writer could find no reference to its origin. Personal communication with Ritchie enlightened the writer on the procedure to be followed.

The method adopted for determining the size-frequency curves is shown in Table 9. Length (L), breadth (B) and elongation ($\frac{L}{B}$) curves are computed in the same manner, thus, a discussion of the elongation size-frequency curves will suffice for all three types.

An x for each grain is placed opposite its appropriate elongation in the table. The x are added up for groups of four consecutive readings, e.g. column III shows that 16 grains measured 1.4, 1.5, 1.6 or 1.7 units and these 16 grains can be considered to approximate the mean value of 1.55 units. In column IV another grouping is used which overlaps the grouping of column III by two units. Both groupings are employed to prepare the curve. As 200 grains were measured, columns III and IV each add up to 200.

The curve is plotted with frequency per cent as the ordinate and elongation as the abscissa (see Figure 4). The values in columns III and IV must be recalculated in terms of percentage so that they may be plotted on the graph, i.e. in column III, the 16 grains representing an average size of 1.55 units actually account for only

$\frac{16}{400} \times 100 = 4$ per cent of the total. This value is plotted opposite the $1.55 \left(\frac{L}{B}\right)$ value on the abscissa. Repetition of this process for each grouping of elongations results in the curves shown in Figure 4.

I	II	III	IV	III A	IV A
$\frac{L}{B}$ MICROMETER SCALE UNITS	FREQUENCY			FREQUENCY PER CENT	FREQUENCY PER CENT
1.2					
1.3					
1.4	xx				
1.5	xxxx				
1.6	xxxxx				
1.7	xxxxxxx				
1.8	xxxxxxx				
1.9	xxxxxxx				
2.0	xxxxxxx				
2.1	xxxxxxxxx				
2.2	xxxxxx xxxxxx				
2.3	xxxxxx xxxxxxxx				
2.4	xxxxxx xxxxxxxx				
2.5	xxxxxx xxxxxx				

4.2	xxx				
4.3	x				
4.4	x				
4.5					
		200	200		
		400			
				100%	

TABLE 9. METHOD OF CALCULATING SIZE FREQUENCY

PLATE 1.

Zircon Euhedra from Sebright M_H Horizon

No. 1.	Actual length	0.22 mm.	Long prismatic euhedra, slightly rounded terminations.
No. 2.	" "	0.23 mm.	Long prismatic euhedra, simple terminations, rod shaped inclusions.
No. 3.	" "	0.26 mm.	Long prismatic euhedra, simple terminations, rod shaped inclusions.
No. 4.	" "	0.23 mm.	Long prismatic euhedra, broken, complex terminations.
No. 5.	" "	0.20 mm.	Long prismatic euhedra, broken, complex terminations, large inclusions.
No. 6.	Actual length	0.20 mm.	Long prismatic euhedra, slightly rounded terminations, note twisting.
No. 7.	" "	0.21 mm.	Long prismatic euhedra, simple terminations.
No. 8.	" "	0.19 mm.	Long prismatic euhedra, simple terminations, note knee twinning.
No. 9.	" "	0.23 mm.	Long prismatic euhedra, simple terminations, note abundance of inclusions and "nibbles" in the side of the crystal.
No. 10.	" "	0.14 mm.	Medium length, broad, euhedra, simple terminations, rod-shaped inclusions.
No. 11.	Actual length	0.25 mm.	Large prismatic euhedra, complex terminations, note "nibbles" and "bar sinister" effect.
No. 12.	" "	0.14 mm.	Medium length euhedra, simple terminations.
No. 13.	" "	0.19 mm.	Medium length euhedra, complex terminations, note large "nibble".
No. 14.	" "	0.17 mm.	Medium length euhedra, complex terminations, note large rod-shaped inclusion.
No. 15.	" "	0.16 mm.	Medium length broad euhedra, simple terminations, note zoning.
No. 16.	Actual length	0.10 mm.	Short stubby euhedra, simple terminations, no inclusions.
No. 17.	" "	0.11 mm.	Short stubby euhedra, simple terminations, no inclusions.
No. 18.	" "	0.11 mm.	Short stubby euhedra, simple terminations, no inclusions.
No. 19.	" "	0.11 mm.	Short stubby euhedra, complex terminations.
No. 20.	" "	0.15 mm.	Medium length euhedra, complex terminations, note large "nibble".

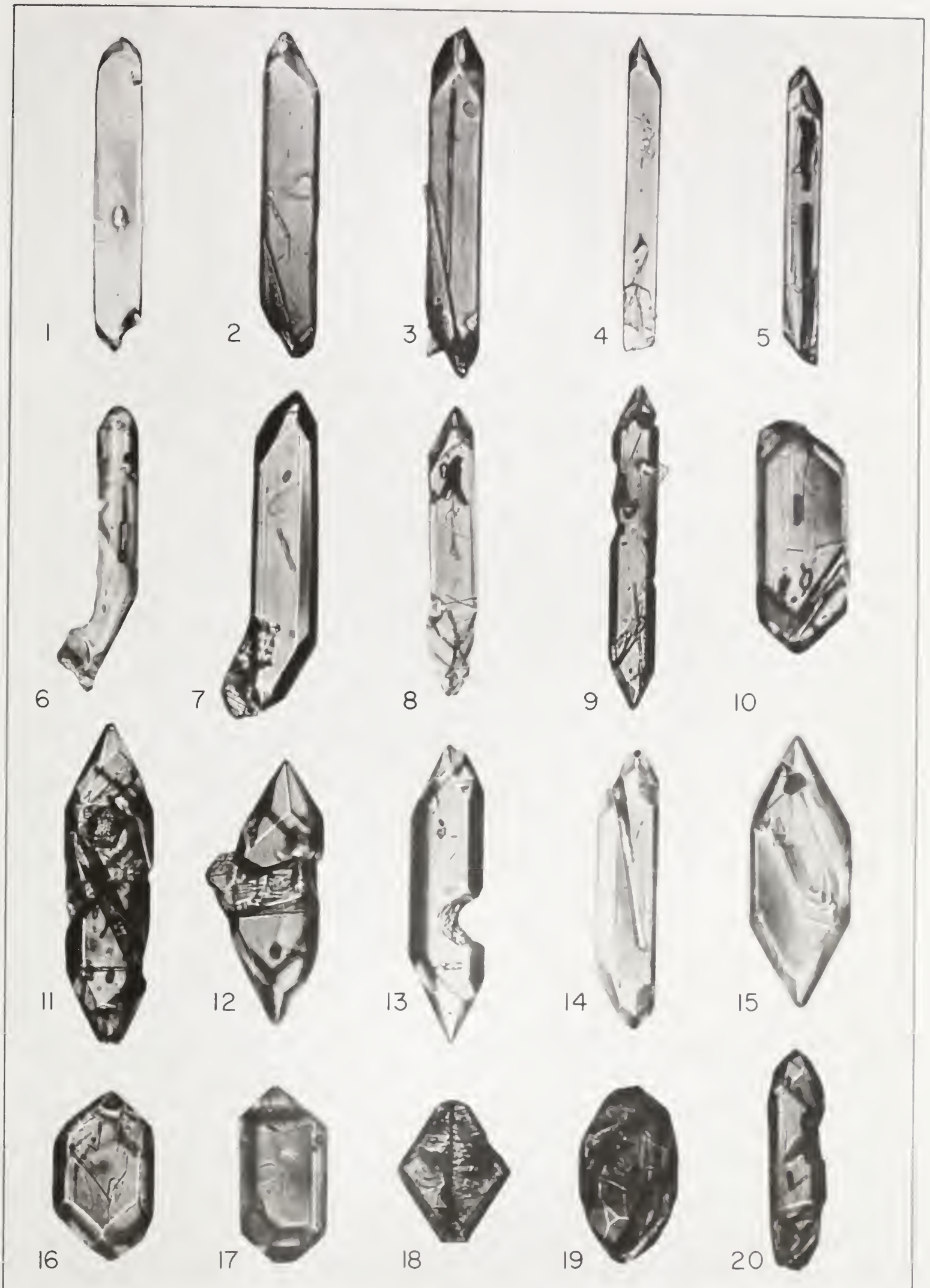


PLATE I.

PLATE 2.

Zircon Euhedra from Medonte M_H Horizon

No. 1.	Actual length	0.27 mm.	Large, broad euhedra, broken, complex termination, note large rod-shaped inclusions.
No. 2.	" "	0.28 mm.	Long prismatic euhedra, broken, simple termination, note variety of inclusions.
No. 3.	" "	0.21 mm.	Long prismatic euhedra, broken, simple terminations, note long inclusion.
No. 4.	" "	0.22 mm.	Long prismatic euhedra, slightly rounded terminations, liquid or gas inclusions.
No. 5.	" "	0.27 mm.	Large, broad, euhedra, broken, complex terminations, note variety and abundance of inclusions.
No. 6.	Actual length	0.21 mm.	Long prismatic euhedra, simple terminations, note large "nibble".
No. 7.	" "	0.18 mm.	Medium length euhedra, slightly rounded terminations, large inclusion.
No. 8.	" "	0.15 mm.	Medium length euhedra, simple terminations, abundant inclusions.
No. 9.	" "	0.18 mm.	Medium length euhedra, simple terminations, note knee twinning.
No. 10.	" "	0.16 mm.	Medium length euhedra, simple terminations, note knee twinning, zoning, large rod-shaped inclusion.
No. 11.	" "	0.13 mm.	Stubby euhedra, simple terminations, note zoning.
No. 12.	" "	0.09 mm.	Stubby euhedra, simple terminations, note overgrowth.
No. 13.	" "	0.09 mm.	Stubby euhedra, simple terminations.
No. 14.	" "	0.14 mm.	Stubby euhedra, simple terminations, note "bar sinister" effect.
No. 15.	" "	0.14 mm.	Stubby euhedra, simple terminations, note zoning and overgrowth.



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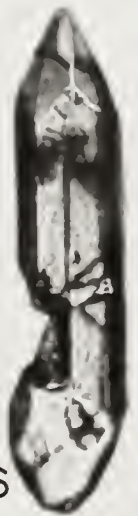
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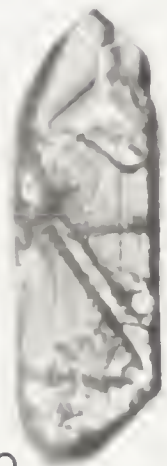
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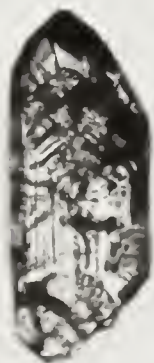
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PLATE 3.

Zircon Euhedra from Coboconk Quarry

No. 1.	Actual length	0.17 mm.	Long prismatic euhedra, broken, complex terminations, note "nibble".
No. 2.	" "	0.19 mm.	Long prismatic euhedra, broken, simple terminations, liquid or gas inclusion.
No. 3.	" "	0.22 mm.	Long prismatic euhedra, broken, simple terminations, rod-shaped and liquid or gas inclusions.
No. 4.	" "	0.16 mm.	Medium length euhedra, broken, simple terminations, abundant rod-shaped inclusions.
No. 5.	Actual length	0.20 mm.	Long prismatic euhedra, broken, simple terminations.
No. 6.	" "	0.18 mm.	Long prismatic euhedra, broken, simple terminations, abundant rod-shaped inclusions.
No. 7.	" "	0.13 mm.	Medium length euhedra, broken, rod-shaped inclusions.
No. 8.	" "	0.15 mm.	Medium length euhedra, broken, complex terminations, large inclusion.
No. 9.	Actual length	0.09 mm.	Short euhedra, broken simple terminations.
No. 10.	" "	0.08 mm.	Short stubby euhedra, simple terminations, note zoning.
No. 11.	" "	0.09 mm.	Short stubby euhedra, simple terminations, note zoning.
No. 12.	" "	0.12 mm.	Short stubby euhedra, simple terminations.



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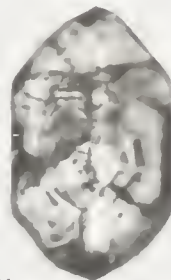
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PLATE 3.

PLATE 4.

Zircon Euhedra from Medonte Mx Horizon

No. 1.	Actual length	0.15 mm.	Rounded grain, rod-shaped inclusion.
No. 2.	" "	0.19 mm.	Long prismatic euhedra, slightly rounded terminations, short rod-shaped inclusions.
No. 3.	" "	0.19 mm.	Well rounded and etched grain.
No. 4.	" "	0.16 mm.	Medium length euhedra, broken, simple terminations, abundant rod-shaped inclusions.
No. 5.	Actual length	0.17 mm.	Broad, medium length euhedra, complex terminations, abundant gas or liquid inclusions.
No. 6.	" "	0.11 mm.	Well rounded grain, large rod-shaped inclusion.
No. 7.	" "	0.11 mm.	Well rounded grain, abundant gas or liquid inclusions.
No. 8.	" "	0.15 mm.	Medium length euhedra, broken, simple terminations.

Zircon Euhedra from Longford M_H Horizon

No. 9.	Actual length	0.10 mm.	Well rounded grain, note zoning.
No. 10.	" "	0.13 mm.	Medium length euhedra, rounded, abundant inclusions.
No. 11.	" "	0.19 mm.	Long prismatic euhedra, simple terminations, note inclusions and "nibble".
No. 12.	" "	0.12 mm.	Short euhedra, rounded, note large dark inclusion.



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PLATE 5.

Apatite from Medonte M_H Horizon

No. 1.	Actual length	0.12 mm.	Stubby euhedra, simple pyramidal terminations, rod-shaped inclusion.
No. 2.	" "	0.15 mm.	Long prismatic euhedra, broken, simple pyramidal terminations, abundant inclusions.
No. 3.	" "	0.19 mm.	Long prismatic euhedra, simple pyramidal terminations.
No. 4.	" "	0.16 mm.	Long prismatic euhedra, simple pyramidal terminations, rod-shaped inclusions, note "nibble".
No. 5.	" "	0.14 mm.	Prismatic euhedra, terminated by pinacoid, large rod-shaped inclusion.

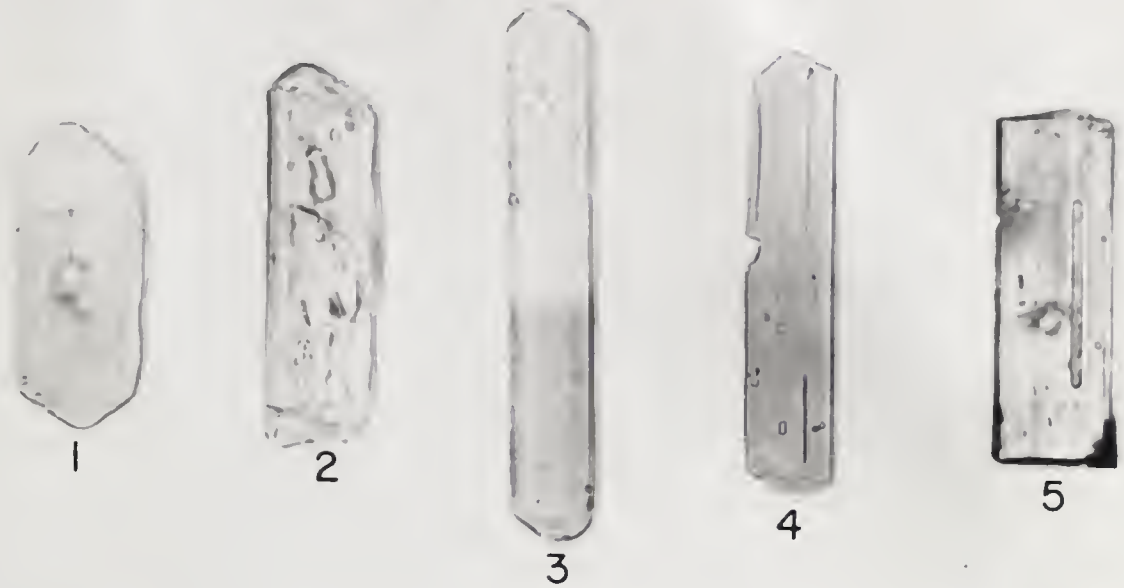
Apatite from Seabright M_H Horizon

No. 6.	Actual length	0.16 mm.	Long prismatic euhedra, terminated by pinacoid.
No. 7.	" "	0.11 mm.	Stubby euhedra, large rod-shaped inclusion.
No. 8.	" "	0.10 mm.	Stubby euhedra, simple pyramidal terminations.
No. 9.	" "	0.12 mm.	Stubby euhedra, etched, large rod-shaped inclusion.
No. 10.	" "	0.15 mm.	Long prismatic euhedra, note variety of inclusions.

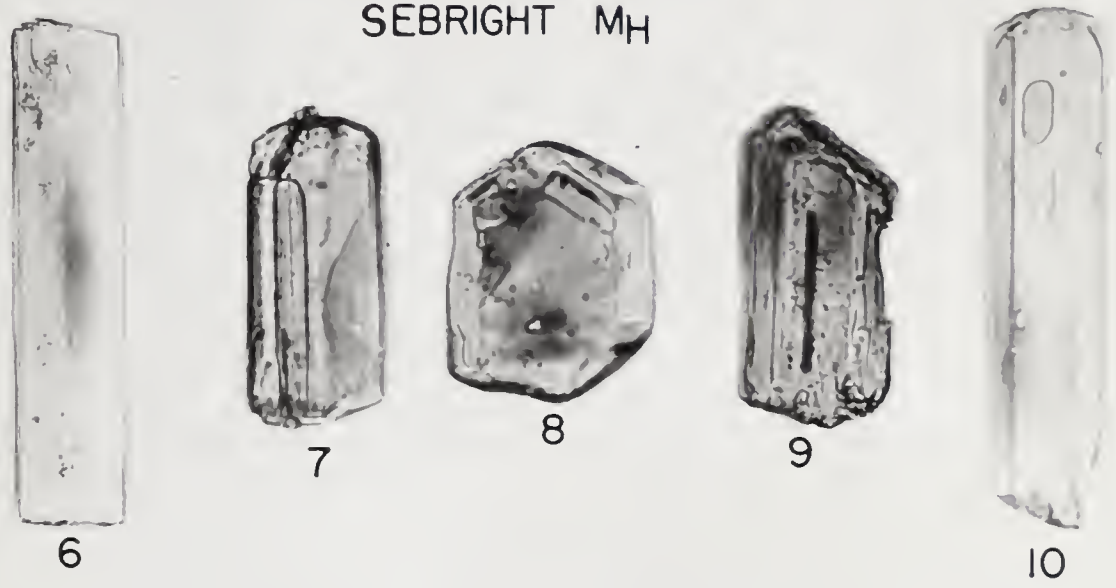
Apatite from Coboconk Quarry

No. 11.	Actual length	0.07 mm.	Stubby euhedra.
No. 12.	" "	0.09 mm.	Prismatic euhedra, etched.
No. 13.	" "	0.12 mm.	Prismatic euhedra, long rod-like inclusion.
No. 14.	" "	0.12 mm.	Slim prismatic euhedra, covered with clay material.
No. 15.	" "	0.11 mm.	Prismatic euhedra, simple pyramidal termination.

MEDONTE M_H APATITE



SEBRIGHT M_H



COBOCONC

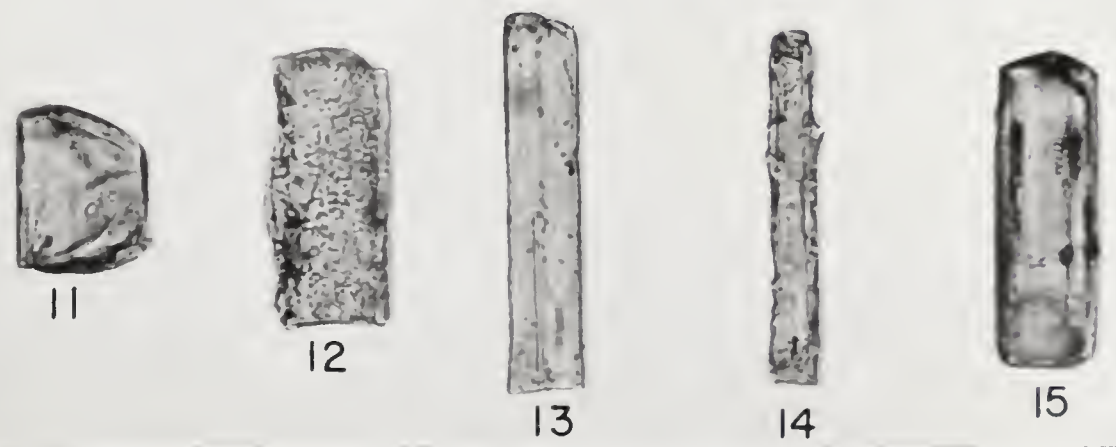


PLATE 6.

Feldspar Fragments from Medonte M_H Horizon

No. 1.	Actual length	0.08 mm.	Sanidine cleavage fragment.
No. 2.	" "	0.10 mm.	Sanidine cleavage fragment. Dark areas are carbonaceous films on cleavage surface.
No. 3.	" "	0.11 mm.	Sanidine fragment.
No. 4.	" "	0.07 mm.	Sanidine cleavage fragment.
No. 5.	" "	0.10 mm.	Sanidine cleavage fragment.
No. 6.	" "	0.08 mm.	Plagioclase cleavage fragment showing etching and carbonaceous films.

Feldspar Fragments from Sebright M_H Horizon

No. 7.	Actual length	0.11 mm.	Sanidine fragment.
No. 8.	" "	0.10 mm.	Sanidine cleavage fragment showing minor etching.
No. 9.	" "	0.13 mm.	Large sanidine cleavage fragment showing minor etching.

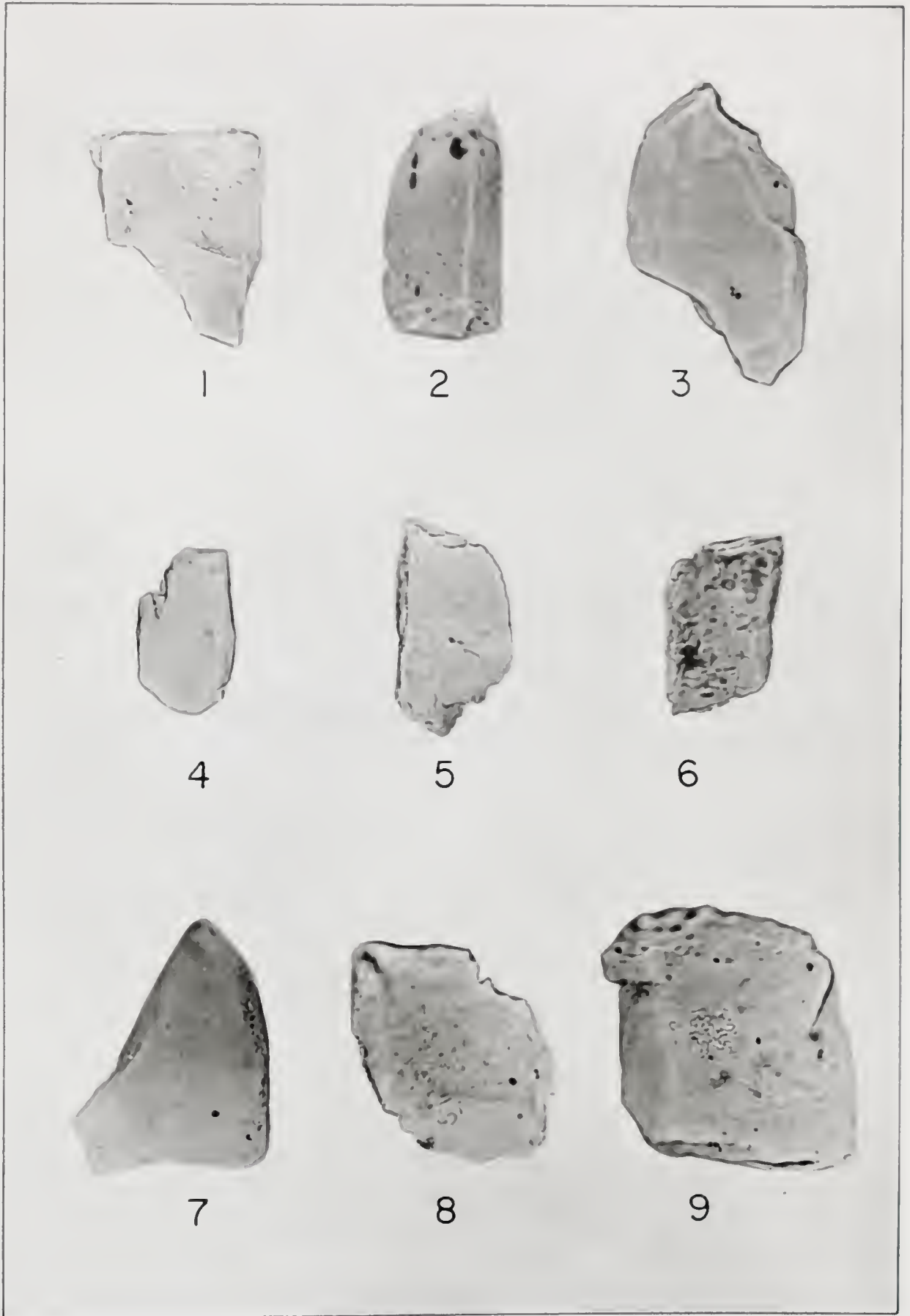


PLATE 6.

PLATE 7.

Feldspar Fields from a Variety of Bentonites

No. 1.	Average grain size	0.08 mm.	Sanidine fragments from Medonte M _H horizon. Dark grains are clay aggregates.
No. 2.	" " "	0.08 mm.	Sanidine fragments from Sebright M _H horizon. Dark grains are clay aggregates.
No. 3.	" " "	0.15 mm.	Sanidine fragments from Kinnekulle bentonite, Type I. Photo after Byström (1956).
No. 4.	" " "	0.08 mm.	Pitted albite grains from Pennsylvanian K-bentonite. Photo after Weaver, (1953).



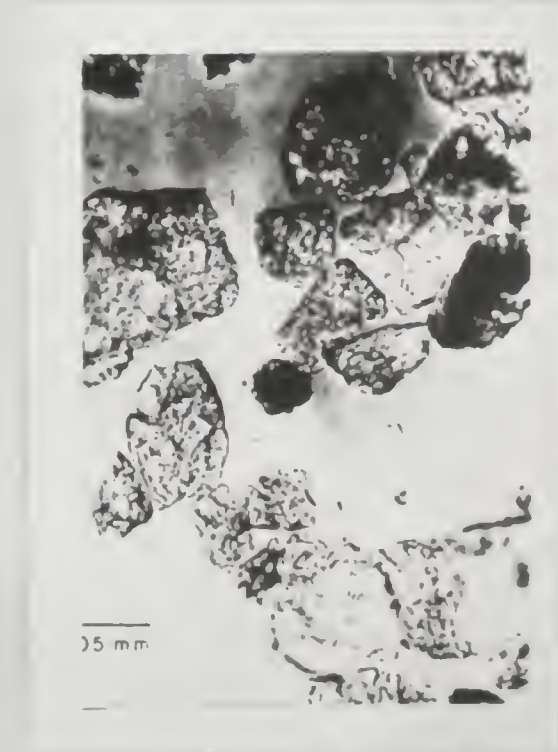
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PLATE 8

Zircon Fields from a Variety of Bentonites

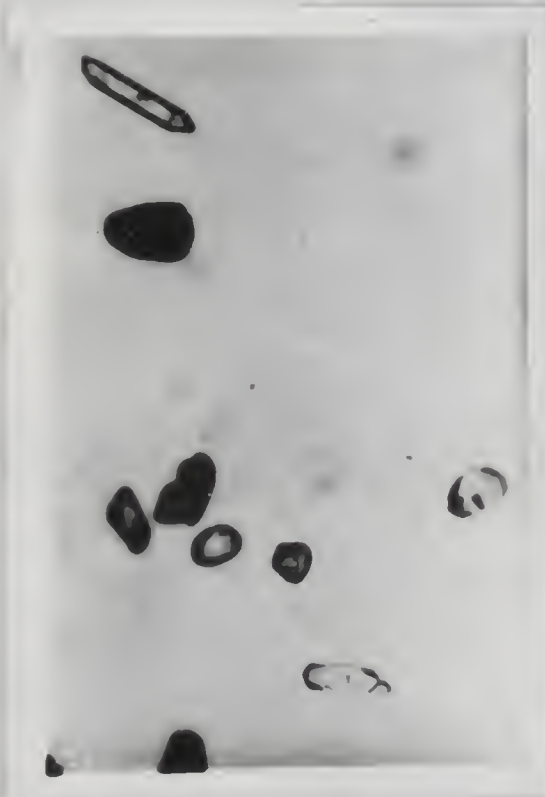
No. 1.	Average grain size	0.14 mm.	Euhedral zircon crystals from Sebright M _H horizon. Opaque grains are pyrite.
No. 2.	" " "	0.14 mm.	Euhedral zircon crystals from Medonte M _H horizon. Opaque grains are pyrite.
No. 3.	" " "	0.11 mm.	Euhedral and rounded zircon grains from Medonte M _x horizon. Opaque grains are pyrite.
No. 4.	" " "		Euhedral zircon crystals from a bentonite horizon within the Martinsburg shale, Fisher's Hill, Virginia. Photo after Carroll (1959).



1



2



3



4

PLATE 9

X-Ray Photographs of Bentonite Clay Fractions

- | | | |
|--------|---|--|
| No. 1. | A.P.I. No. 38. | Potash Bentonite from Moccasin
Formation (Middle Ordovician) Catawaba,
Virginia. |
| No. 2. | A.P.I. No. 41. | Potash Bentonite from Moccasin formation
(Middle Ordovician) Tazewell, Virginia. |
| No. 3. | Bentonite from Medonte M _H horizon. | |
| No. 4. | Bentonite from Sebright M _H horizon. | |
| No. 5. | Bentonite from Longford M _H horizon. | |
| No. 6. | Bentonite from Medonte M _x horizon. | |
| No. 7. | Bentonite from Coboconk quarry. | |

1.



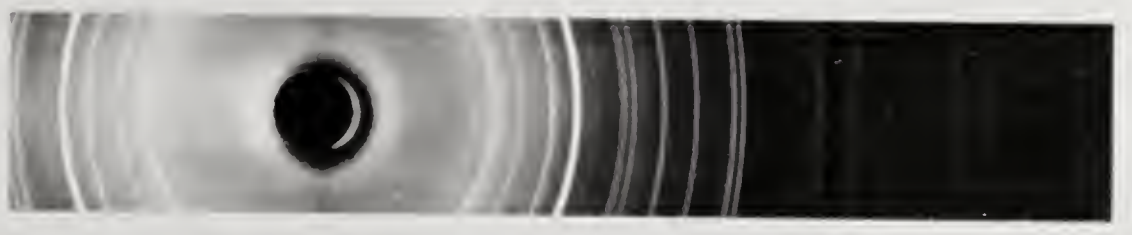
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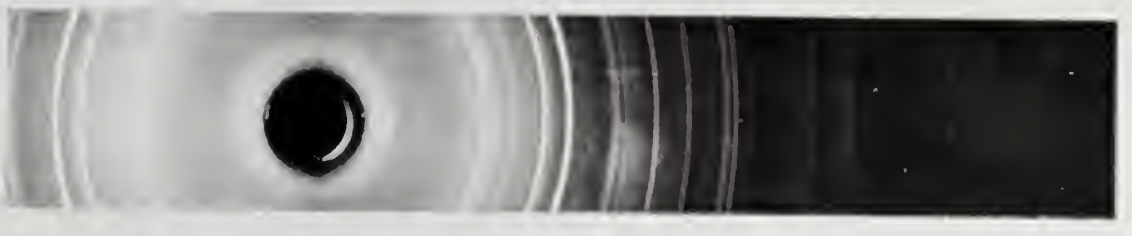
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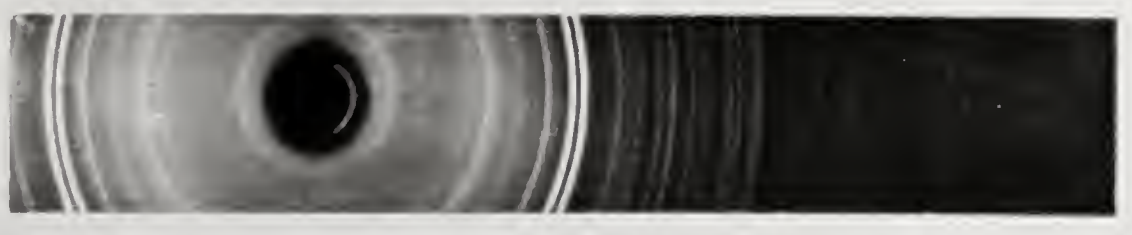
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